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INDEX TO SUBJECTS.

A.

	PAGE
Abnormal samples of milk, C. W. HEATON and others ...	1 to 6
Acetone, Volumetric estimation of, ROBINEAU & ROLLIN ...	148
„ The solubility of rosin and mineral oils, or their mixtures, in, F. WIRDEBROLD ...	206
Acidimetric and alkalimetric solutions, Standardizing of, PARSONS, ...	95
Acido-butyrometry, N. GEBBER ...	137
Acts, Defects in the Food Adulteration ...	35
„ Suggested improvements in the working of the Food ...	37
Adulteration Acts, On the results of the working of, in Dublin, SIR C. CAMERON ...	213
Alcohols, The detection of monovalent, B. V. BITTO ...	146
„ Methyl and ethyl, C. A. LOBBY DE BRUYN ...	136
Alkaline liquids containing hypochlorite, Titration of, C. ULLMANN ...	255
Alumina and ferric oxide in mineral phosphates, Determination of, MARIANI & TABELLI ...	90
Alumina and ferric oxide, Separation of, H. BOENTRAGER ...	146
Ammonia, Presence of, in zinc dust, ROBINEAU & ROLLIN ...	144
Ammonia, The Nessler reaction for, B. NEUMANN ...	232
Ammonium thiocyanate in manures, P. L. JUMEAU ...	135
Analysis, Electrolytic methods of, OETTEL ...	151
Analytical Chemists, Formation of Italian Society of ...	21
Aniline oil, The analysis of, H. REINHARDT ...	150
Annual meeting of the Society ...	25
Annual address, President's ...	25
Annual dinner ...	45
Antimony from arsenic, Separation of, L. GARNIER ...	259
Apparatus for the estimation of insoluble fatty-acids, C. E. CASSAL ...	44
Apparatus, New distilling, for the Stock nitrogen process, W. F. K. STOCK ...	58
Apparatus for the estimation of free and albuminoid ammonia in water-analysis, A. H. GILL ...	65
APPOINTMENTS:—	
Mr. John White to West Bromwich ...	24
Mr. W. W. Fisher, as District Agricultural Analyst for the County of Oxford ...	308
Appointments, Joint, of Medical Officer of Health and Public Analyst undesirable ...	31
Arsenicalis liquor, New volumetric method for the valuation of, S. GRÖNY ...	259
Arsenic from antimony, Separation of, L. GARNIER ...	259

	PAGE
Ash in mineral lubricating oils, Determination of, A. STEPANOW ...	94

B.

Babcock machine, On some modifications of, C. R. C. TICHBORNE ...	237
Basic slag, The determination of lime in, A. F. HOLLEMAN ...	21
Beer, Boric acid a natural constituent of, J. BRAND ...	135
„ Detection of saccharin in, F. GANNTER ...	184
Beeswax, The examination of, G. BUCHNER ...	233
Boiling-point of salt solution, H. D. RICHMOND ...	142
Boric acid a normal constituent of hops and beer, J. BRAND ...	135
Boric acid, On a method of titrating, R. T. THOMSON ...	184
Boric acid, Methods for the determination of, A. K. REISCHLER ...	222
Borofluoride of potassium, Acidimetric estimation of, F. STOLBA ...	282
Boron, The determination of, H. MOISSAN ...	254
Butter, Average composition of, during 1892 ...	52
„ The volatile fatty acids of, H. KREIT ...	134
„ Mixtures of, with margarine, or other fats, The recognition of, A. M. HOUZEAU ...	204
„ Note on a new method of examining, C. E. SOHN ...	218
„ Distinguished from margarine, F. GANNTER ...	284
„ The content of leicithin in, E. WRAPPELMAYER ...	305
Butter analysis, comparison of Reichert-Wollny and Leffmann-Beam processes ...	55
„ „ On the (so-called) Penne- tier's method of, A. PIZZI ...	91
„ „ by polarized light, A. PIZZI ...	91
„ „ Use of cryotile fibre in ...	77
„ „ Kreis' modification of the Reichert-Meissl process ...	145
„ „ sulphuric acid as a hydro- lyzing agent, S. RIDEAL ...	165
Butter-fat, Sulphuric acid hydrolysis of, S. RIDEAL ...	165
Butyric acid, The distillation of, H. D. RICHMOND ...	17

C.

“Caccio-Cavallo,” The analysis of, G. SARTORI ...	17
Cadmium from copper, Separation of, by the iodide method, P. E. BROWNING ...	304
Canada, Particulars as to public analysts in	86
Carbolic acid, Assay of crude, G. SCHACHEL ...	48

	PAGE
Castor-oil, Oxidized or soluble	226
Centrifugal milk-testers, Note on, P. VIETH	201
Centrifugal separator, Rapid determination of crude fibre by, W. THÖRNER	138
Centrifugal milk-testers unfitted for use by unskilled persons	202, 237
Apparatus for heating, R. H. WILKINSON	292
Certificates on adulterated samples, Note on, A. W. STOKES	287
Cheese, The analysis of "Caccio-Cavallo," G. SARTORI	17
" Chemical researches on Sicilian, SPICA & DE BLASI	94
" Use of crysotile fibre in the analysis of	77
" Manufacture of, from sheep's milk, C. BESANA	248
Chlorides, A source of error in estimating by Mohr's method, W. S. YOUNG	125
Chlorine in water, Note on the estimation of, F. FAIRLEY	222
Chromium in ferro-chromium, The determination of, SPÜLLER & KALMANN	251, 252
Cod-liver oil, Acidity of, due to use of old livers	197
Coffee, Use of crysotile fibre in the analysis of	81
Coffee glazing, F. FELSINGER	147
Condensed milk, Points in the analysis of, RICHMOND & BOSELEY	170
Consistency of semi-solids, Simple appliance for testing the, C. E. SOHN	218
Copper sulphate, On the examination of, J. RUFFLE	279
Copper from cadmium, separation by the iodide method, P. E. BROWNING	304
Copper sulphate, Rapid detection of iron in, G. GRIGGI	305
CORRESPONDENCE:—	
Letter from Mr. A. W. Stokes	24
from Dr. Morgan	187
" from Messrs. Stokes and Bodmer	212, 260
" from Messrs. Richmond and Boseley	236
" from Mr. W. F. K. Stock	90
Cotton-seed-oil in lard, Detection of, F. GANNTER	183
" " Detection of heated, W. G. CROOK	221
Cream, Average amount of fat in, during the year 1892	52
" Rising of, in churns during delivery	54
Crysotile fibre, On the use of, in proximate organic analysis, T. MACFARLANE	73
D.	
Definitions of vinegar, Various	180
Drugs, Unsatisfactory condition of the analysis of	29
Dublin, On the results of the working of the Adulteration Acts in, SIR C. CAMERON	218

	PAGE
E.	
Electrolytic methods of analysis	151
Electrolytic separations, The use of definite voltages in, H. FEENDENBERG	257
F.	
Fat, Note on an abnormal melting-point of, E. J. BEVAN	286
Fat in the milk of Mecklenburg herds, Amount of, P. VIETH	301
Fats and oils, Use of crysotile fibre in the analysis of	78
Fats, Hydrolysis of, by concentrated sulphuric acid, PRAGER & STERN	230
Fats by strong sulphuric acid, Saponification of, SCHATZMANN & KREIS	304
Fatty acids, Estimation of insoluble, C. E. CASSAL	44
Fatty oils, The oxidation of, W. FAHRION	283
Ferric oxide and alumina in mineral phosphates, Determination of, MARIANI & TASSELLI	90
Ferro-chromium, The determination of chromium in, SPÜLLER & KALMANN	251, 252
Ferric oxide and alumina, Separation of, H. BOENTRAGER	146
Fibre, Rapid determination of, crude by centrifugal separator, W. THÖRNER	138
Food Act of Dr. Cameron, Strictures on the Fowler's solution, New volumetric method for the valuation of, S. GYÖRY	30, 259
G.	
Gas dissolved in potable waters, Observations on the methods in use for the determination of, G. MUSAIO	67
Ginger, with special reference to the discrimination between genuine and "exhausted" specimens, DYER & GILBEAD	197
Glue, Valuation of leather, F. GANNTER	284
Gunpowder, Use of crysotile fibre in analysis of	83
H.	
Hops, Boric acid a natural constituent of, J. BRAND	135
Hydrazines with lignin, Reactions of, E. NICKEL	283
Hydrolysis of fats by concentrated sulphuric acid, PRAGER & STERN	230
Hydroxylamine, Quantitative precipitation in presence of, JANNASCH & MAI	255
Hypochlorite, Titration of alkaline liquids containing, C. ULLMANN	255
I.	
Indiarubber surrogates, Contributions to the chemistry of, R. HENRIQUES	223
Indiarubber goods, The analysis of, R. HENRIQUES	227
Indiarubber goods, The analysis of, D. HOLDE	147
Indiarubber, Recovered, R. HENRIQUES	258
Inland Revenue, Extract from the report of the Commissioners of	259

	PAGE
Iodine, Qualitative examination for cyanogen in, C. MEINKE	93
Iron pyrites, and oxide of iron in mineral phosphates, Distinction between, H. H. B. SHEPHERD	261
Iron in commercial copper sulphate, Rapid method for the detection of, G. GRIGER ...	305
L.	
Lard, The analysis of steam, A. GOSKE ...	18
" The analysis of, AMTHOR & ZINK ...	20
" Detection of cotton-seed-oil in, F. GANNTER	183
" Detection of heated cotton-seed-oil in, W. J. CROOK	221
Lead, Estimation of oxygen in, LUNGE & SCHMID	68
LEGAL:—	
The sale of baking-powder containing alum	152
Is coloured acetic acid vinegar?	208
"Substance, Nature, and Quality." Important decision	306
Leffmann-Beam method of estimating fat in milk, H. D. RICHMOND	130
Leicithin in butter, The content of, E. WRAMPPELMYER	305
Lignin, Reactions of hydrazines with, E. NICKEL	283
Lime, Determination of, in basic slag, A. F. HOLLEMAN	21
Lister-Babcock milk-testers, Suggestions for the extended use of, G. EMBREY ...	118
M.	
Maize-oil, Notes on, A. SMETHAM ...	191
Malt liquors, Use of crysotile fibre in the analysis of	79
Manganese, The determination of by means of hydrogen peroxide, A. CARNOT ...	231
Manures, Ammonium thiocyanate in, P. L. JUMEAU	135
Margarine, The adulteration of, with sunflower-oil, JOLLES & WILD	232
Meeting, Report of special, of Society of Public Analysts, Feb. 15, 1893	97
Melting-point, Note on an abnormal, E. J. BEVAN	586
Methyl alcohol, A more stable body for many purposes than ethyl alcohol, L. de BRUYN	136
Methylene blue as a reagent for sugar in urine, N. WENDER	301
Microscopic examination of butter by polarized light, A. PIZZI	91
Milk, Abnormal samples of	275
" " C. W. HEATON ...	1
" " B. DYER ...	2
" " A. SMETHAM ...	3
" " H. D. RICHMOND ...	4
" " W. F. LOWE ...	6
" Amount of fat in, of Mecklenburg herds, P. VIETH	301
" Changes in frozen	53, 56

	PAGE
Milk, Points in the analysis of condensed, RICHMOND & BOSELEY	170
" Definition of genuine	7-12
" Distinction between skimmed and separated	173, 175, 176
" Note on the detection of the adulteration of fresh with diluted condensed mil	174
" Phosphates in, DUCLEUX	149
" Presence of nitrates in, as a test for adulteration	272, 279
" Ritthausen's method of estimating casein and albumin in	172
" Sebelien's method of estimating casein and albumin in (foot-note)	171
" Sheep's, and the manufacture of cheese therefrom, C. BESANA	248
" Size of globules of fat as a test for adulteration in	271, 273, 276
" The action of heat upon, RICHMOND & BOSELEY	141
Milks, The discrimination between normal and adulterated, H. D. RICHMOND ...	270
Milk analysis, Average of 23,000 samples during year 1892	51
" " Comparison of Adams' and Werner-Schmid process	53
" " Estimation of total solids by Babcock method	54
" " Use of crysotile fibre in	74
" " Lister-Babcock tester, G. EMBREY	118
" " Leffmann-Beam method of fat estimation, H. D. RICHMOND	130
" " acido-butyrometry, N. GERBER	187
" " strength of sulphuric acid in	119, 123
Milk and milk products, The composition of, H. D. RICHMOND	50
Milk sugar, The action of heat upon, RICHMOND & BOSELEY	141
Milk-testers, New apparatus for heating centrifugal, R. H. WILKINSON	292
" Centrifugal, not fitted for the use of unskilled persons	202
" Note on centrifugal, P. VIETH	201
Mineral oils, The determination of fatty oils in, A. KLIMONT	146
Molasses, Use of crysotile fibre in the analysis of	81
Mustard, Use of crysotile fibre in the analysis of	83
N.	
Nitrates, The direct determination of nitrogen in, A. DEVADA	92
" The determination of nitrogen in, by reduction, T. F. SCHMITT ...	144
Nitric acid, Determination of, O. F. ROBERTS ...	305
Nitrogen, New method for the rapid determination in organic substances, W. F. K. STOCK	

INDEX.

	PAGE
Nitrogen, Modified distilling apparatus for the Stock process, W. F. K.	
Stock	58
„ Estimation of, by sulphuric acid and chromic acid in organic substances	62
„ in nitrates, Direct determination of, A. DEVADA	92
„ in urine, Rapid method of estimating, PETIT & MONFED ...	148
Nitrous and nitric acid in sulphuric acid, Amount of	262

O.

OBITUARIES :—

Davies, Mr. R. H.	285
Heaton, Mr. G. W.	237
Officers of the Society, Election of	43
Oil, The solubility of rosin and mineral, and mixtures of, in acetone, F.
WIEDERHOLD	206
„ Soluble or oxidized castor	226
„ The analysis of sperm, containing mineral oil, C. A. L. DE BRUYN	281
„ Almond, Possible adulteration with oils from the kernels of apricots, cherries, plums, etc., C. MICKÉ	149
„ Olive, Researches in the adulteration of, V. OLIVIERA	302
Oils, Determination of ash in mineral and lubricating, A. STEPANOW	94
„ and fats, Use of crysotile fibre in the analysis of	78
„ The determination of fatty, in mineral	146
„ Notes on maize and rice, A. SMETHAM	191
„ Action of sulphur chloride on	225
„ The oxidation of fatty, W. FAHRION	283
Olive oil, Researches in the adulteration of, V. OLIVIERA	302

P.

Pepper, Use of crysotile fibre in the analysis of	83
Phenacetin, Qualitative examination of, C. PLATT	303
Phosphorus, New method for the determination of yellow, J. TORH	256
Phosphates in milk, DUCLAUX	149
Phosphates, Distinction between iron pyrites and oxide of iron in the commercial analysis of mineral, H. H. B. SHEPHERD	261
Potassium boro-fluoride, The acidimetric estimation of, F. STOLBA	282
Public analysts in Canada, Particulars as to	86

R.

Reichert process, Notes on the theory and practice of the, H. D. RICHMOND	64
Reichert-Meisel process, Kreis modification of	144
Report of special meeting of Society of Public Analysts, Feb. 15, 1898	98
Report of the Commissioners of the Inland Revenue, extract from	259
Results in water-analysis, The interpretation of, W. F. STODDART	293

REVIEWS:—	PAGE
A guide to the examination of the urine, J. WICKAM LEGG	308
Analysis of milk and milk-products, LEFF- MANN & BEAM	284
Catalogue of scientific apparatus and re- agents, BRADY & MARTIN	22
Die chemischen Unterschiede zwischen Kuh und Frauen Milch und die Mittel zu ihrer Ausgleichung, F. SOXHLET ...	139
Experiments on magnesia alba, etc., J. BLACK	235
Galenic pharmacy, R. A. CRIPPS	140
Gli Olii, DR NEGRI & FABRIS	207
Modern microscopy, CROSS & COLE ...	235
Olii vegetali, animali e minerali, G. FABRIS	23
Public Health Laboratory work, KENWOOD & BOYCE... ..	162
Reactions: a selection of organic prepara- tions important to pharmacy in regard to their behaviour to commonly-used reagents, F. A. FLÜCKIGER	307
The coal-tar colours, THEODORE WEYL...	23
The principles of theoretical chemistry, IRA REMSEN	71
Traité général d'analyse des beurres, A. J. ZUNÉ	22
Traité pratique d'analyse chimique à de recherches toxicologiques, G. GUÉRIN ...	185
Untersuchungen aus der Praxis der Gährungsindustrie, C. E. HANSEN ...	72
Rice-oil and maize-oil, Notes on, A. SKE- THAM	191
Ritthausen's method of estimating proteins in milk	172
Rubber goods, Analysis of, H. HENRIQUES ...	223
" " " D. HOLDE	147
Rubber, Analysis of recovered, H. HEN- RIQUES	258

S.

Saccharin in beer, Detection of, F. GANNTE	184
Salt solutions, Boiling-point of, H. D. RICHMOND	142
Salts, On the solubility of difficultly soluble, A. F. HOLLEMAN	257
Sand fraudulently mixed with seeds, The determination of, G. CUGNI	251
Saponification, Rapid, for analytical purposes, A. SMITHAM	193
Saponification of fats by strong sulphuric acid, SCHWATZMAN & KRIS	304
Saponification equivalent, Confusion concerning foreign	196
Sebelien's method of estimating albumin and casein in milk (foot-note)	171
Separations, Use of definite voltages for electrolytic, H. FREUNDENBERG	257
Sheep's milk and the manufacture of cheese therefrom, C. BESANA	248
Soap, Use of crysolite fibre in the analysis of Sodium oxide to analysis, The application of, W. HEMPEL	205

	PAGE
Solubility of difficultly-soluble salts, A. F. HOLZEMANN	257
Somerset House, Unsatisfactory relation between public analysts and	32
Somerset House and water-analysis, F. W. STODDART	246
Sperm-oil containing mineral oil, The analysis of, C. A. L. DE BRUYN	281
Spirite, Use of crysotile fibre in the analysis of	79
Steam-lard, The analysis of, A. GOSKE	18
Sulphur, Critical examination of methods for the estimation of, L. MARCHLEWSKI	282
Summer meeting	189
Sulphuric acid as the saponifying agent in butter analysis	145
Sulphuric acid as the saponifying agent in butter analysis, S. RIDEAL	165
Sulphuric acid, strength of in relation to specific gravity (foot-note)	123
Sulphurous acid in wine, M. RIPPER	69
Syrups, Use of crysotile fibre in the analysis of	81
T.	
Tartar emetic, New method for the valuation of, S. GYÖRY	259
Tartaric acid, determination of free, in wines, L. SOSTEGNI	91
U.	
Urine, Rapid process for the determination of organic nitrogen in, PETIT & MONFET	148
" Methylene blue as a reagent for sugar in, N. WENDER	301
V.	
Vinegar, ALLEN & MOOR	180, 240
" Addition of sulphuric acid to	(foot-note) 182
" Official strengths of	183

	PAGE
Vinegar, Prejudicial effect of albuminoids on keeping properties of,	241
" From calcium acetate	242
" Analysis of Essigspirit	242
" Sulphuric acid in	243
" Remarks on methods of analysis of, and interpretation of results	244 to 246
Vinegars, analysis of various	241, 242, 243, 246
" Strength of various	243

W.

Water, Note on the estimation of chlorine in, T. FAIRLEY	222
Waters, Observations on the methods in use for the determination of the quantity of gas dissolved in, G. MUSAIO	67
Water analysis, Apparatus for the estimation of free and albuminoid ammonia in, A. H. GILL	65
" " Source of error in estimating chlorine volumetrically, W. G. YOUNG	125
" " Dr. Dupré's method of viewing solution in oblique estimation	129
" " the interpretation of results, F. W. STODDART	293
" " and Somerset House, F. W. STODDART	246
Wine, Aldehyde sulphite in, M. RIPPER	69
" Determination of free tartaric acid in, L. SOSTEGNI	91
" Some points in the determination of the colour of, L. SOSTEGNI	91
" Sulphurous acid in, M. RIPPER	69

Z.

Zinc dust, Presence of ammonia in, ROBINEAU & ROLLIN	144
--	-----

INDEX OF AUTHORS' NAMES.

	PAGE
A.	
ALLEN, A. H. & G. H. MOOR, Vinegar	180, 240
AMTHOR & ZINK, Analysis of lard	20
B.	
BESANA, C., Sheep's milk and the manufacture of cheese therefrom	248
BEVAN, E. J., Note on an abnormal melting-point	286
BITTO, B. V., Detection of monovalent alcohols	146
BOENSTRAGER, H., The separation of ferric oxide and alumina	146
BRAND, J., Boric acid as a normal constituent of hops and beer	185
BROWNING, P. E., The separation of copper and cadmium by the iodide method	304

	PAGE
BRUYN, LOBBY DE, The analysis of sperm-oil containing mineral oil	281
BRUYN, LOBBY DE, C. A., Methyl and ethyl alcohols	136
BUCHNER, G., The examination of beeswax	233
C.	
CAMERON, SIR CHARLES, On the results of the working of the Adulteration Acts in Dublin	213
CARNOT, A., The determination of manganese by hydrogen peroxide	231
CASSAL, C. E., Estimation of insoluble fatty acids	44
CROOK, W. G., The detection of heated cotton-seed oil in lard	221

	PAGE
CUGINI, G., On the determination of sand fraudulently mixed with seeds ...	251
D.	
DEVADA, A., The direct determination of nitrogen in nitrates ...	92
DYER, BERNARD, B.Sc., Abnormal sample of milk ...	2
DYER & GILBARD, Ginger, with special reference to discrimination between genuine and "exhausted" samples ...	197
DUCLAUX, Phosphates in milk ...	149
E.	
EMBEEY, G., The Lister-Babcock milk-tester, and some suggestions for extending its use ...	118
F.	
FAHRION, W., The oxidation of fatty oils ...	283
FAIRLEY, T., Note on the estimation of chlorine in water ...	222
FELSINGER, F., Coffee glazing ...	147
FRENDENBERG, H., The use of definite voltages for electrolytic separations ...	257
G.	
GANNTER, F., Detection of cotton-seed-oil in lard ...	183
" Detection of saccharin in beer ...	184
" Valuation of leather glue ...	284
" Butter distinguished from margarine ...	284
GARNIER, L., Separation of arsenic and antimony ...	259
GERBER, N., Acido-butyrometry ...	137
GILL, A. H., Apparatus for the estimation of free and albuminoid ammonia in water ...	65
GOEKE, A., The analysis of steam-lard ...	18
GRIGGI, G., Rapid method for the determination of iron in commercial copper sulphate ...	305
GYÖRY, S., New volumetric method for the valuation of Fowler's solution and of tartar emetic ...	259
H.	
HEATON, C. W., Abnormal sample of milk ...	1
HEMPFL, W., The application of sodium peroxide to analysis ...	205
HENRIQUES, R., The analysis of rubber goods ...	13
" Contributions to the chemistry of indiarubber surrogates ...	223
" The analysis of rubber goods ...	227
" Recovered rubber ...	258
HOLDE, D., The analysis of rubber goods ...	147
HOLLEMAN, F., The determination of lime in basic slag ...	21
" Solubility of difficultly soluble salts ...	257
HOUEFAU, A. M., On the recognition of	

	PAGE
mixtures of butter with margarine or other fats whether animal or vegetable ...	204
J.	
JANNASCH & MAI, Quantitative precipitation in presence of hydroxylamine ...	255
JOLLES & WILD, The adulteration of margarine with sunflower oil ...	232
JUMEAU, P. L., Ammonium thiocyanate in manures ...	135
K.	
KLIMONT, J., Determination of fatty oils in mineral oils ...	146
KREIS, Modification of the Reichert-Meissl process ...	145
KREIT, H., The volatile fatty acids of butter ...	134
L.	
LOWE, W. F., Abnormal sample of milk ...	6
LUNGE & SCHMID, Estimation of oxygen in lead ...	68
M.	
MACFARLANE, THOMAS, On the use of crysotile fibre in proximate organic analysis ...	73
MARCHLEWSKI, L., Critical examination of methods for the estimation of sulphur ...	282
MARIANI & TASSELLI, Determination of ferric oxide and alumina in mineral phosphates ...	90
MEINKE, O., Qualitative examination of iodine for cyanogen ...	93
MICKO, C., The oils from the kernels of apricots, cherries, peaches, and plums, as possible adulterants of almond oil ...	149
MOISSAN, H., The determination of boron ...	254
MUSATO, G., Observations on the methods in use for the determination of the quantity of gas in potable waters ...	67
N.	
NEUMANN, B., The Nessler reaction for ammonia ...	---
NICKEL, E., Reactions of hydrazines with lignin ...	283
O.	
OTTTEL, On electrolytic methods of analysis ...	151
OLIVIERI, V., Researches on the adulteration of olive oil ...	302
P.	
PARSONS, Standardizing alkalimetric and acidimetric solutions ...	95
PETIT & MONFET, Rapid process for determining organic nitrogen in urine ...	148
PIZZI, A., On the (so-called) Penmetier's method of butter analysis (microscopic examination by polarized light) ...	91
PLATT, C., Qualitative examination of phenacetin ...	303
FRAGKE & STERN, Hydrolysis of fats by concentrated sulphuric acid ...	230
R.	
REINHARDT, H., The analysis of aniline oil ...	150
REISCHLER, A. K., Methods for the determination of boric acid ...	232

	PAGE		PAGE
RICHMOND, H. DRÖPF, Abnormal samples of milk ...	4	wines, and in the determination of free tartaric acid ...	91
" The distillation of butyric acid ...	17	SPICA & DE BLASI, Chemical researches in Sicilian cheese ...	94
" The composition of milk and milk-products ...	50	SPÜLLER & KARLMANN, The determination of chromium in ferro-chromium ...	251, 252
" Note on the theory and practice of the Reichert process ...	64	STEPANOW, A., The determination of ash in mineral lubricating oils ...	94
" The Leffmann-Beam method of estimating fat in milk ...	130	STOCK, W. F. K., A modified distilling apparatus for the determination of nitrogen by the Stock process ...	58
" The boiling point of salt solutions ...	142	" The Stock method for the rapid determination of nitrogen in organic bodies ...	58
" The discrimination between abnormal and adulterated milks ...	270	STODDART, F. W., Somerset House and water analysis ...	246
RICHMOND & BOSELEY, Note on the detection of adulteration of fresh milk with diluted condensed milk ...	174	" Water analysis—the interpretation of results ...	293
Points in the analysis of condensed milk ...	170	STOKES, A. W., On the form of certificates on adulterated samples ...	287
" " " The action of heat on milk ...	141	STOLBA, T., The acidimetric estimation of potassium borofluoride ...	282
RIDEAL, S., The sulphuric acid hydrolysis of fats ...	165	T.	
RIPPER, M., Sulphurous acid in wine ...	69	THOMSON, R. T., On a method of titrating boric acid ...	184
ROBERTS, C. F., Determination of nitric acid ...	305	THÜRNER, W., Rapid determination of crude fibre by centrifugal separator ...	138
ROBINEAU & ROLLIN, The presence of ammonia in zinc dust ...	144	TICHBOENE, C. R. C., On some modifications of the Babcock machine ...	237
" " " Volumetric estimation of acetone ...	148	TOTH, J., A new method for the determination of yellow phosphorus ...	256
RUFFLE JOHN, On the examination of copper sulphate ...	279	U.	
S.		ULLMANN, C., Titration of alkaline liquids containing hypochlorite ...	255
SARTORI, G., Some analyses of Cacciocavallo, (cheese) ...	17	V.	
SCHACHERL, G., The assay of crude carbolic acid ...	48	VIRTH, PAUL, Note on centrifugal milk-testers ...	201
SCHATZMANN & KREIS, Saponification of fats by strong sulphuric acid ...	304	" The amount of fat in the milk of Mecklenburg herds ...	301
SCHMITT, T. F., The determination of nitrogen in nitrates ...	144	W.	
SHEPHERD, H. H. B., The distinction between iron pyrites and oxide of iron in the commercial analysis of mineral phosphates ...	261	WENDER, N., Methylene blue as a reagent for sugar in urine ...	301
SMITHAM, A., Abnormal samples of milk ...	3	WIEDERHOLD, F., The solubility of rosin-oil and mineral oils, or mixtures thereof, in acetone ...	206
" Notes on rice-oil and maize-oil ...	191	WILKINSON, R. H., New form of apparatus for heating the water in centrifugal milk-testers ...	292
" On rapid saponification for analytical purposes ...	193	WRAMPPELMEYER, E., The content of leicithin in butter ...	305
SOHN, C. E., Simple appliances for testing the consistency of semi-solids, with a note on a new method of examining butter ...	218	Y.	
SOSTEGNI, L., Some points in the determination of intensity of the colour of		YOUNG, W. GAWTHORNE, On a source of error in estimating chlorides by Mohr's method ...	125

THE ANALYST.

JANUARY, 1893.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The usual Monthly Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday, December 7th, the President (Mr. O. Hehner), occupying the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed :—as Members, A. F. Morgan, F.C.S., Newport, Mon.; C. G. Moor, B.A., F.C.S., 101, Leadenhall Street, E.C.; T. S. Tweedie, Annan; —as Associate, J. F. Liverseege, F.I.C., Birmingham.

Messrs. G. Watson Grey and George Thomas Holloway were duly elected members of the Society.

The following papers were read and discussed.

“On the use of Crysolite Fibre in Proximate Organic Analysis.”

By THOMAS MACFARLANE.

“On the Leffmann-Beam Process.” Part II.

By H. DROOP RICHMOND.

Mr. C. G. Moor exhibited some novel and ingenious forms of laboratory apparatus, consisting of a retort stand, a gas generator, a mouth blowpipe, and a chromic acid battery.

This concluded the proceedings of the Society.

VARIOUS PAPERS ON ABNORMAL MILK.

(Read at the Meeting, November 2nd, 1892.)

No. 1, by C. W. HEATON.

On August 9th, 1892, I received from the Inspector of the Parish of St. Martin's-in-the-Fields, a sealed sample of milk. He stated that his deputy had seen the milk

drawn that morning from the udder of a cow. The results of analysis were as follows :—

Specific gravity	1031·43
Solids, non-fatty	8·39 per cent.
Fat	·26 „
Total	8·65 „
Ash	·79 „
Chlorine in Ash	·20 „
The cream separated in the lactometer				
was	1·00 „

The sample was, of course, obtained by partial milking, and according to law it could not be said to be adulterated.* Yet, what analyst, in ignorance of the facts, could possibly certify that such a milk was genuine?

This case seems to me to illustrate the inefficiency of the Food and Drugs Act in its present form. Surely, it would be better to insert in a schedule, the minimum strength which was to be held as representing genuine milk. The vendor could, of course, dilute or skim his milk down to this standard, but that is abundantly possible now. Milk below a certain strength ought not to be sold without special description.

It must be remembered that, to quote the words of the twelfth report of the Local Government Board, “the Sale of Food and Drugs Acts are not designed to prevent the sale of poor articles, but that of adulterated articles.”

No. 2, by B. DYER, Sc.D.

During the milking trials at the Essex Agricultural Society's Show at Harlow, this year, the writer met with a sample of abnormal milk which was of some interest. The cow yielding it was under four years old. She was milked dry on the eve of the show in the presence of the writer's colleague, Mr. Rosling. On the next morning she was again milked dry, and the morning milk was weighed and sampled for analysis. It weighed 16½ lbs. It was observed at the time of milking, that the cow was restless and nervous, and the attendant who milked her observed that she “held up” some of her milk. The milk when analysed gave :—

Total solids	10·85
Fat	1·85
Solids-not-fat	9·00
Ash	0·77

* It was decided, so far back as 1877, that milk should be sold “whole.” A fine of £10 was inflicted in a case where the defendant had sold “fore” milk for genuine milk. (THE ANALYST, vol. i., p. 82). —S.—Edit.

On the afternoon of the second day, being curious to investigate further a case in which milk of such abnormal quality was yielded by a cow in good health and condition, the writer had her milked out again. This time the milk was normal and of good quality, shewing :—

Total solids	12.75
Fat	3.64
Solids-not-fat	9.11

The milk obtained at the trial milking was like partially skimmed milk, and it is to be presumed that the strange deficiency of fat was due to the "holding up" of the richer part of the milk. It is well known that the "strippings" are richer in fat than the main bulk of the milk, but not that so great a proportion of fat is liable to be withheld, as was the case here.

The main interest is centred in the fact that the cow normally yielded good normal milk; and the case serves to show that even an appeal to the cow may be sometimes misleading. Not only should a cow be milked "dry," but she should be milked carefully and without any unusual excitement, if a correct sample of her yield is required.

No. 3, by A. SMETHAM.

For several years past it has been the custom of the Altrincham Agricultural Society to award prizes at its annual show for the best cow for dairy purposes, and in order to estimate the relative merits of the animals exhibited, the judges have awarded points for the appearance (frame, etc.) of the cows, and have then left the final award to me, the award to be made by granting points for appearance of cow, quantity of milk produced and for the weight of the butter-fat and of solids-not-fat yielded during the trial.

This year four cows were entered for competition, and below I give the figures relating to each :—

No.	Weight of Milk. lbs. ozs.	Total Solids. per cent.	Fat. per cent.	Ash. per cent.	Sp. Gr.
545	16 10	14.88	5.73	.68	1.0319
546	8 4	12.79	3.31	.80	1.0347
547	6 6	19.50	11.06	.53	1.0266
548	13 6	16.06	7.37	.72	1.0278

The cows were milked dry at eleven o'clock in the morning in the presence of the stewards, and they were again milked for the purposes of adjudication at 4 o'clock in the afternoon. It was the milk obtained in the second milking which I examined.

Although I was not present when the samples were taken I am assured that care was observed to prevent any tampering with the milk, and that, after weighing, the samples were immediately bottled in presence of the judges and forwarded to me. Indeed, the conditions under which the trials take place, practically preclude the

possibility of any tampering, for the milking is performed in view of the public as well as of the judges and stewards. It is also in the interest of each exhibitor to obtain as much milk as possible from his cow, as points are given for quantity as well as quality.

All the cows exhibited were shorthorns, but the only one which gave abnormal milk was the one numbered 547 in the catalogue. This was a three-year-old non-pedigree shorthorn, which had had her first calf three weeks before the show and had been liberally fed on a compound feeding cake and bean flour in addition to her ordinary rations. The milk marked No. 548 was from a cow which had had her second calf two months previous to the show, while that marked No. 545 was from a cow which had only calved one week.

In considering these results it must be remembered that the milk was obtained from four picked cows from one of the best dairy districts in England, and that the conditions of a show yard are not such as conduce to a normal flow of milk; but, nevertheless, they are interesting as showing within what limits pure milk from shorthorns may vary.

No. 4, by H. D. RICHMOND.

A sample of the evening milk from a farm in the West of England had the low specific gravity of 1·0300; this caused a suspicion of watering, but on analysis the figures found were:—

Specific gravity	1·0300
Total solids	12·32
Fat	3·70
Solids-not-fat	8·62

From that date for the succeeding five weeks a sample was analysed from every churn received from this farm.

The weekly averages are as follows:—

Week Ending.	Morning Milk.					Evening Milk.			
	Sp. Gr.	T.S.	Fat.	S.-N.-F.		Sp. Gr.	T.S.	Fat.	S.-N.-F.
Aug. 20	... 1·0312	12·31	3·68	8·63		1·0298	12·64	4·07	8·57
„ 27	... 305	12·30	3·84	8·46		297	12·56	4·18	8·38
Sept. 3	... 310	12·47	3·91	8·56		303	12·90	4·41	8·49
„ 10	... 310	12·59	3·92	8·67		306	12·75	4·15	8·60
„ 17	... 305	12·30	3·68	8·62		302	12·94	4·26	8·68

Of the total of 160 samples, no less than 16 fell below 8·45 per cent. of solids-not-fat, and a further 14 fell between 8·45 per cent. and 8·5 per cent. of solids-not-fat.

The milk was supplied by a herd of 60 cows, who yielded an average quantity of 60 gallons a day, a very small quantity for the time of year.

On the 27th of August the whole of the cows were milked in the presence of witnesses, and samples were taken, as soon as the cows were milked, after the milk was cooled, and on the arrival of the milk in London; the analysis of these three series of samples differed only in the second place of decimals; the results were:—

			Sp. Gr.	T.S.	Fat.	S.-N.-F.
17 cows	1.0300	12.32	3.97	8.35
24 „	305	12.80	4.21	8.59
19 „	295	12.40	4.12	8.28

As these samples were strictly authenticated, and the possibility of water being added is precluded, I have no hesitation in accepting the other low samples as genuine; that they were so was further shown by the practical identity of the figures for milk-sugar, casein, albumen and ash in the authenticated and unauthenticated low samples.

The average of seven analyses during the week ending August 27th, was:—

Water	87.25
Fat	4.32
Milk-sugar	4.21
Casein	2.91
Albumen42
Salts77
				99.88

At the time when the low samples occurred, the cows were out to grass, and, owing to the bad season the pastures were extremely bare; I recommended an addition of saccharine matter to the food, and this was commenced in the week ending September 3rd, and in the week ending September 10th three samples were submitted to analysis with results as follows:—

Water	87.49
Fat	3.91
Sugar	4.43
Albuminoids	3.40 (difference)
Salts77
				100.00

The average solids-not-fat had risen from 8.42 per cent. in the week ending August 27th to 8.63 per cent. in the week ending September 10th, or a rise of .21 per cent.; the sugar had concurrently risen from 4.21 per cent. to 4.43 per cent., a rise of .22 per cent.

Other samples from farms in the same district have been found as low as 8.4 per cent. in solids-not-fat, and a similar deficiency in sugar has been found.

In all the samples received from this farm the fat calculated by means of the milk-scale has been found to be about .2 per cent. in excess of that actually estimated; a

deficiency in sugar, lowering the average density of the solids-not-fat, would partially account for this, but the difference is greater than would be due to the deficiency found. As Béchamp (*Comptes Rendus*, 1873, 2d. Semestre, 436) has described alcohol as a normal constituent of milk, I attempted to estimate this. After making 100 c.c. alkaline with baryta and passing a current of carbonic acid to remove the excess, I distilled 20 c.c. and found the density of the distillate to be 1·00005 and 1·00007 in two experiments; alcohol, then, if present at all is not in sufficient quantity to account for the low specific gravity.

Vieth (*THE ANALYST* xiv. 69.) has drawn attention to Recknagel's observation that the specific gravity of milk rises for some hours after milking, and has supplemented his researches; the average rise is ·0013; it is possible that this rise had for some reason not taken place in the milk from this farm.

From my analyses of milk this year, I can draw the following conclusions:—

i. During the months of June, July, August, and September of this year, the solids-not-fat have been fully ·1 per cent. below the average.

ii. This deficiency is due to a lowness in the amount of milk-sugar, and is probably attributable to the condition of the pastures due to the bad season.

iii. Authenticated samples have given as little as 8·28 per cent. of solids-not-fat (average milk of 19 cows); two unauthenticated samples having been lower than this, viz.: 8·22 per cent. and 7·92 per cent.

iv. During these four months the fat calculated by means of the milk-scale has been in excess of that estimated, the average of results (excluding this abnormal farm) has been ·07 per cent. too high. [This farm has been excluded as the great number of samples would destroy a true average.]

No. 5, by W. F. LOWE.

This milk was taken August 17th 1881, from a cow 9 years old and apparently in very good condition. The cow was milked in my presence and was milked dry, at the usual time of milking. The cow had calved at Christmas, and it was receiving a daily feed of Indian meal and cotton cake on coming up to be milked. The milk had an unpleasant saline taste, especially when cold. It yielded the following results:—

				I.	II.
Total Solids	8·82	8·83
Fat	2·84	2·73
Solids-not-fat	5·98	6·10
Ash	·93	·95
Chlorine in Ash...	·266	·268

The cow was some-time afterwards killed for food and was said to be healthy.

DISCUSSION.

Mr. A. J. de Hailes drew attention to the results of analysis of certain samples examined by him. He had found

Specific Gravity	1026·00	1028·00	1026·50	1031·00	1031·50
Total Solids	11·90	11·33	10·27	14·54	13·98
Fat	4·17	3·45	2·80	4·77	3·92
Solids-not-fat	7·73	7·88	7·47	9·77	10·06
Ash	0·59				

In all these cases Mr. de Hailes stated he had seen the cows milked. He subsequently stated that he put them on the board because they were abnormal samples.

In reply to a question put by Mr. H. Droop Richmond, Mr. de Hailes stated that the gravity was taken with a specific gravity bottle.

Mr. Cassal thought that while the discussion was of undoubted importance to the Society, it went to show the necessity for the re-consideration by the council, or, perhaps, by the whole of the Society, of the definitions given to the articles dealt with by public analysts under the Sale of Food and Drugs Acts. Everything depended upon definitions. Mr. Heaton had contributed a note referring to what he called "poor but genuine milk." He (Mr. Cassal) would like to ask Mr. Heaton, Mr. Richmond, or any who made a speciality of milk analysis, what they meant when they said "genuine milk." The only definition with which he was acquainted was this,—that "milk is a liquid yielded by the lacteal glands of the cow." He did not consider that this was a definition which could possibly be accepted by scientific men. It ought surely to be obvious that the lacteal glands of a cow could be made to yield all sorts of remarkable liquids. By ill-treating a cow, by starving her, by improperly feeding or poisoning her, that cow could be made to yield various things, but those things were not milk. He had had samples of so-called milk, containing considerable amounts of pus and blood. Would Mr. Heaton say that a liquid consisting partly of pus and blood was "genuine milk" because it was extracted from the udder of a cow? He declined to accept the analysis of Mr. de Hailes, or analyses such as that of which Mr. Heaton had given details, as representing, in any sense, the composition of the article known as "milk," simply because it was alleged that the fluids analysed had been extracted from the udders of one or more abnormal cows. He invited the serious attention of the Society to the desirability of laying down a scientific plan upon which to work, based upon the many thousands of analytical results which had been obtained in reference to the natural product in question, and which showed its normal composition to vary between certain limits. It was well known within what limits natural milk varied and what was the "nature, substance, and quality" of that which the purchaser had a right to get when he asked for milk, and he contended that it should be laid down in any future Act of

Parliament relating to the adulteration of food, that, taking milk as an example, in cases where samples of so-called milk differed in composition from the well-known normal composition of milk, and were therefore not of the "nature, substance, and quality" of milk, it should be incumbent upon the person who sold these articles to prove that they had been produced in a legitimate manner. Rational definitions to be applied to other articles of food would also have to be agreed upon. The matter was one of vital importance.

Mr. de Hailes agreed with Mr. Cassal's remarks with reference to definitions. In his opinion there was no branch of trade in which such a rule of thumb method obtained, as in the milk trade. Everything that came from the udder of a healthy cow was called milk. Why should there not be two varieties of milk? The first variety could command the higher price, and the inferior could be sold at a lower price.

Dr. John Muter said he had always understood that, supposing a certain process were used—say the paper-coil process, or something which was known to be equally good—commercial mixed milk was a substance which should contain not less than $11\frac{1}{2}$ per cent. of total solids, and 3 per cent. of total fat. This seemed to him to be a very good definition of milk. He agreed with Mr. Cassal, and did not see that it followed, because an analyst occasionally received abnormal samples, that such definition should be affected. For one sample of an abnormal character (supposing all the cows in England were milked at one time), there would be thousands and thousands above the standard; the solitary exception only went to prove the rule. He objected to people bringing up extraordinary analyses for the purposes of defence in adulteration cases, as was occasionally done. A man, for instance, might go into Court and swear that he once analysed a so-called milk, or even two or three so-called milks, with extraordinary results; and he might give evidence for the purpose of defeating another public analyst who had worked on the recognised standard; and this, most unfortunately, was sometimes done. Instead of correcting blunders, such a man was trying to undo all the standards and to give milk dealers in England the power of adulterating milk to a certain extent, depending on the standard of abnormality. The Society should not take official notice of odd or abnormal samples. The question was—what were the constituents of average normal milk in England? He believed that the present standard of the Society could not be improved upon.

Mr. Allen concurred with Mr. Cassal and Dr. Muter. He thought that the figures brought forward in the various papers had a purely academic interest, since no public analyst would think of condemning samples of milk having the composition of the abnormal samples described. It had also been laid down very clearly by Mr. C. Estcourt, in a paper read some years ago before the Society, that the solids-not-fat should not be the sole criterion in judging of milk, but that other data should also be taken into account. From the point of view of the administration of the Act, the results recorded

by the authors of the paper were almost without significance at any rate, there was no fear of injustice being done to vendors. He thought that gentlemen who had found abnormal figures were quite justified, when called for the defence, in stating candidly that they had occasionally met with some samples of such composition which they believed to be genuine: but it was a pity that they did not go on to tell the *whole* truth, and say that such samples were of such rarity that the probabilities in favour of the milk in question being adulterated enormously out-weighed, by, perhaps, 10,000 to 1, the remote chance of its being genuine, and that the analyst who had an opportunity of examining the milk when perfectly fresh was more likely to be right than those who examined it at a later period. It was a great scandal that the Court should often be misled by evidence of the kind, and wrong-doers should consequently escape.

Mr. de Hailes, in reply to a question put by Mr. Alfred H. Allen, stated that the samples had not been taken from a herd of cows, but from single animals.

Mr. A. W. Stokes referred to the results of analysis of some milks yielded by a herd of thirty-one cows during three months.

The President asked Mr. Stokes whether he had any personal knowledge of how the samples were taken.

Mr. Stokes said he had not seen the cows milked.

Mr. Stokes thought, until some legal standard could be fixed, it was unwise to publish the results of confessedly abnormal samples. These were used in law courts as samples of genuine normal milks. Even in Dr. J. Bell's book on food not a single reference was made to the many hundreds of thousands of milk analyses made by other analysts on ordinary samples, but the pages of *THE ANALYST* have been ransacked to find every recorded abnormal analysis. It might be well for the Society to revise, in full committee, the present standards, and to invite all those who had experience of well-authenticated cases differing from the normal to detail them, but not for publication. There is a feeling in the trade, even by those who wish to raise the standard, that occasionally the innocent suffer.

Mr. H. Droop Richmond could not agree with some of the remarks made by Mr. Stokes, namely, that there was a general feeling in the trade that the standards of analysts were too high. Several representative meetings of the trade had been held, and the unanimous opinion expressed at these was that the standards of the Society of Public Analysts were too low; they admitted of a very considerable quantity of separated milk being mixed with and sold as genuine milk, a practice which was largely adopted by unscrupulous dealers; and the general sense of the meetings was that the standards should be raised, in order to prevent this fraud being committed. He (Mr. Richmond) worked with a high standard, and he found that nearly all his milks came up to it, and he made complaint in those rare instances in which they did not do so, with the result that the quality soon improved. He believed it possible that in the cases mentioned by

Mr. Stokes the cooler may have leaked, and this was possible without the knowledge of anyone. As Mr. Stokes had no means of satisfying himself on this point, his *very numerous* low samples could not be considered as authenticated, even if the reputation of a farmer were considered sufficient guarantee of the genuineness of milk which, perhaps, passed through half-a-dozen different hands. Such *genuine* low samples might suit the unscrupulous dealers before referred to, but they would not bear looking at from an impartial point of view.

Mr. Stokes remarked that if, as Mr. Richmond said, the cooler leaked, it only did so in the morning and never in the afternoon, through the three months' series of analyses.

Mr. Richmond drew attention to the fact that the low gravity of Mr. de Hailes' samples was an abnormality. He was under the impression that there was only one definition of milk—the *normal* secretion of the mammary glands of the cow. He thought some definition, such as the one he had given, which was a reasonable one should be inserted in the Sale of Food and Drugs Act. He also thought that a clause should be inserted to the effect that all milks below a certain composition should be considered suspicious, and the onus of proving the genuineness of such milks should be borne by the vendor, as had been previously suggested by another speaker. He would consider it sufficient proof of a milk being normal if the cows, milked on some convenient date, as soon after the samples were taken as possible, yielded milk below the Society's standard.

Mr. Alfred H. Allen said that in Manchester a system was carried out according to which, when a milk was found adulterated, the vendor was informed of the fact, and invited to allow an Inspector to visit his farm and see the cows milked, with a view to taking another sample of milk, and comparing it with that previously analysed.

Dr. Bernard Dyer said that the quotation of cases of abnormal milk should be of no use at all in defending cases under the Act. In bringing his note forward he did so as an illustration of what even a normal cow in normal health, under certain conditions might do, and to show that even an appeal to the cow herself might be fallacious or misleading if made under unusual conditions, as, for instance, if she were milked by strange hands or in presence of a group of strange witnesses.

Mr. E. J. Bevan thought it would be a matter of considerable interest if those gentlemen who had analysed such abnormal milks would tell the Society whether, in the future, if such samples came under their notice, they proposed to regard them as adulterated or not. It seemed to him that, as those gentlemen who were taking part in the discussion were speaking to a Society of public analysts and not one of milk vendors, such a question was not at all an improper one to ask.

Mr. Benedict Kitto said that his predecessor was sent a sample of milk one day and he returned it as having had so much water added. The Inspector believed the analyst

in that case to be wrong, because the farmer declared—and there were circumstances surrounding the case which showed the probable truth of the statement—that the milk was not watered. Of another sample sent after a short interval, the analysis was returned as being that of a very poor milk, but probably genuine. In the latter case the Inspector was tolerably certain that the milk was watered, because when he took the sample the man was reluctant to serve him, whilst in the former case it was just the reverse. The reason for the poorness of the milk in the first case given by the vendor in Court was that the cow had been affected by a blizzard, a period of most severe weather which lasted for 10 days or longer; and, after hearing Dr. Dyer's statements as to the great differences in the quality of the milk yielded on two successive days by a cow at a cattle show, he could conceive that this farmer's cow might have been so affected by the sudden blizzard that it gave an abnormally poor milk on this particular occasion.

The President remarked, as Mr. Smetham had very pertinently observed, an animal placed in a show, or under conditions that were likely to excite it, could not be expected to yield normal milk. He considered, therefore, that important conclusions could not be drawn from some of the samples referred to that evening. Dr. Dyer had given a very cogent explanation as to the reason for the abnormality of the milk which he had analysed, namely that the cow was excited, and yielded, on one occasion, exceptional milk. In Mr. Richmond's figures the differences from normal milk were so small that no cautious Public Analyst would have declared the samples to be adulterated. Not so, as regards Mr. Heaton's sample. He (the President) would have liked to have had full information concerning this sample. They knew only that it was sent by an Inspector, who stated that the Sub-Inspector had seen the cow milked. Coming to the results of Mr. Lowe it seemed evident that the cow was diseased. The ash was altogether out of proportion to the solids-not-fat, and Mr. Wynter Blyth had observed this in certain diseases. No doubt it was very interesting that diseased cows might yield such a fluid, but he could not see how this concerned them as Public Analysts. He would like to ask Mr. de Hailes in what condition were the cows, the milk of which he had referred to? It was an extraordinary thing, that in one single herd there should be so many cows yielding milk below the ordinary standard. Exceptional samples were undoubtedly sometimes met with that point had been agreed upon years ago—but exceptions surely were not so very common that from a herd of 30 cows many of the animals were found to yield milk of the nature described by Mr. de Hailes. Analysts had experience of immense numbers of milk and he (Mr. Hehner) could not but look upon the sudden appearance of so many abnormal samples with a great deal of suspicion.

He had already expressed his surprise at some remarks that had been made by Mr. Stokes. He thought that a Public Analyst who brought matters before them which affected both the general public, and the agricultural interests of the whole country, would most carefully weigh his remarks. He was astonished to hear Mr. Stokes bring

forward his results and then admit that the samples upon which the remarks were based were furnished to him by someone else who was, or might be, deeply interested in having these, to say the least, suspicious samples passed as genuine milks. They had from Dr. Vieth and Mr. Richmond the experience of the Aylesbury Dairy Company, where a process of considerable selection was going on, and they found that not one sample in 100,000 examined fell below, to any notable extent, the limit adopted by this Society; while Mr. Stokes asserted, that such exceptions were not only not rare, but frequent. By careful selection of the cows a great deal could be done, and he believed it was the duty of Public Analysts to teach the public and farmers to select their cows so that the public got a fair value for their money. It had been shown that if Public Analysts condemned milk with less than 8.5 per cent. of solids-not-fat, such milk disappeared from the market, and he believed, if they insisted upon 8.6 or even 8.8 they would soon get it, while if they always attached so much importance to the so-called lowest milk, they were not doing their duty to the public who had no one but the Public Analysts to advise them as to what milk ought to be. If a person purchased milk, he expected to obtain milk of fair composition and quality. The milk-consumer is distinctly prejudiced if the milkman sold milk containing only 8.0 per cent. of solids-not-fat. He refused to accept such a fluid as genuine milk. As regards other articles to which Mr. Cassal had referred, such as butter, he did not see how at present any definition of composition from a chemical point could be made. He could not see, with his present knowledge of the subject, how anyone who obtained a smaller proportion of soluble or volatile fatty acid in his butter was prejudiced, as the quality of butter did appear to be independent of the composition as regards soluble and insoluble fatty acids. He did not therefore think that a rigid definition of butter could at present be fixed upon. But in the case of milk the urgency for such a definition was very great. He thought Dr. Muter's remarks on this subject were much to the point. A limit by which milk should be judged had been carefully fixed by the Society and had since been confirmed by tens of thousands of analyses. It was certainly well for Public Analysts to know that there were occasional exceptional milks, but they should not help in depressing the limits upon the basis of occasional abnormal samples. For a Public Analyst to make the most of these abnormal milks, to hold them up as the milks by which the whole milk-supply should be judged and regulated appeared to him a grave scandal. Such persons were counteracting the good done by the general body of Analysts.

At the same time it should be distinctly understood that Public Analysts had absolutely no interest to get up cases against milkmen. Abnormal milks were the products of abnormal animals, and must always be considered in the light of their experience as exceptional and rare. They could be of no influence upon the mixed milk of herds of cows as sold by milkmen and used by the public.

The Analysis of Rubber Goods. R. Henriques. (*Chem. Zeit.*, xvi., 1892, 1595, 1596, and 1623, 1626, and 1644, and 1645.—The extreme difficulty of making a satisfactory analysis of rubber articles is well known, and the author has accordingly endeavoured to arrive at methods which, although not professing to solve all the problems presented, go a good way towards removing existing stumbling blocks. In the first place, the sampling of rubber goods is not easy to effect, as commercial articles are not homogeneous. For example, in a sheet containing much siliceous matter, at one place as little as 1.4% of SiO_2 was found, and in another no less than 28%. In the case of soft rubber goods, therefore, the sample must be cut into tiny cubes, and these well mixed, and fairly large quantities taken for analysis; while with hard rubber, division by means of a rasp is a convenient plan. The utility of an elaborate analysis of the ash of manufactured rubber has been much overrated, as on account of the alterations undergone by the mineral substances originally present during the process of incineration, the nature of these additions can with difficulty be deduced from the composition of the ash, whether obtained by simple incineration or by subsequent treatment with ammonium nitrate, carbonate and other reagents, designed to bring it into a uniform condition. The determination of the inorganic constituents of rubber is best effected in conjunction with the estimation of the total sulphur. Methods for the determination of the latter are numerous, but the author having tried the recognised dry methods and the plan of dissolving the rubber in an oxidising solution, such as nitric acid, has adopted a combination of the two. It is carried out as follows:—A small, but fairly deep, porcelain basin is covered with a funnel with a wide stem cut off short, and 20 c.c. of pure fuming nitric acid placed in it. Three to four grams of the rubber, cut into small pieces, are then gradually introduced through the stem of the inverted funnel, the operator waiting for the diminution of the reaction induced by each piece before putting in another. After solution has been completed, the dish is cautiously warmed on the water-bath, as a good deal of frothing may take place, the solution evaporated to a syrupy consistency, and the treatment with the acid repeated more than once if necessary. During the most violent part of the action of the acid the funnel stem may be covered with another smaller funnel, to prevent loss by spirting. Finally, to the concentrated solution is added 4 grams of a mixture of three parts of nitrate of potash and four of carbonate of soda, the whole warmed for some time on the water-bath and then fused in the basin, still covered by the funnel. Care must be taken that the melt be not too strongly alkaline, as otherwise the basin will be unduly attacked. When graphite is present in the rubber, prolonged fusion is inevitable in order to ensure its complete oxidation. The melt is treated with dilute hydrochloric acid, evaporated to dryness to render the silica insoluble, and taken up again with dilute nitric acid. Should the whole go into solution, it can be made up to a known volume, a portion taken for the determination of the sulphur, and the rest used for the estimation of the inorganic constituents of the rubber by the ordinary methods. In the contrary case, the residue can only consist of silica, barium sulphate and lead

sulphate. The latter is dissolved out with ammonium acetate, added to the main filtrate, and the analysis of the latter proceeded with as before. The silica and barium sulphate are separated by the usual means, and then, since barium sulphate has been recognized in the residue, the presence of barium in the filtrate will be a conclusive proof that the whole of the sulphur was left in the residue; and, *vice versa*, the presence of sulphur in the filtrate leads directly to the deduction that it cannot contain barium, and the work of analysis is by so much shortened. When the rubber contains little ash, and the total sulphur only is wanted, the quantity taken for analysis need not exceed 0.25 or 0.5 grms., and two evaporations with nitric acid, followed by fusion as described above, suffice. Carius' method is also available, but is, in the author's opinion, unsuited for the requirements of technical analysis.

Besides the total sulphur, it is important to know what quantity of the element is present as sulphur used for vulcanising, as distinct from that present as sulphides and sulphates. The process of dissolving the rubber in oil of turpentine has many drawbacks, and is, at the best, inexact. The following is the plan adopted by the author in its place. Commercial kerosene is shaken with caustic soda to remove sulphur compounds, dried and distilled, the fraction coming over between 140° and 250° being collected. 5 grams of the finely divided rubber, or 10 to 12 grams if it be low in ash, are placed in a weighed 250 c.c. flask, about 150 c.c. of purified kerosene added, and the flask fitted with a condensing tube and heated in an oil bath to 140°, to 150°, CO, until the pieces of rubber have lost all coherence and the insoluble portion is left as a powder at the bottom. In order to make certain that the temperature has not been so high as to lead to the formation of sulphuretted hydrogen from the action of the sulphur on the kerosene, a second flask containing sulphur, and a similar quantity of the same kerosene, is heated in the same bath as the flask containing the rubber, and the evolution of sulphuretted hydrogen watched for by inserting a piece of lead paper in the neck. When solution is complete, the flask is put aside in a moderately warm place to allow the insoluble portion to settle, the solution poured through a weighed filter, the residue washed a few times by decantation, brought upon the filter, and the flask washed out with hot petroleum, without special pains being taken to remove the whole of the insoluble matter from its walls. The washing is completed with petroleum ether of low boiling point, and the flask and filter dried together at 110°C. and weighed. There is now no difficulty in determining the amount of the sulphur in the insoluble residue and the form in which it is present, and this value deducted from that of the total sulphur already determined, gives the value for the free and vulcanising sulphur. One point needs notice. It is possible that when the mineral matter incorporated with the rubber consists partly or entirely of oxidising bodies, such as red lead, or of metallic oxides or carbonates, a portion of the vulcanising sulphur may become changed in the rubber itself into sulphates, the author having found small quantities of calcium sulphate in a rubber containing lead oxide and chalk. It is not possible to tell in such a case whether the calcium sulphate has been

added intentionally or has formed from the vulcanising sulphur. The residue from the petroleum treatment is also available for the direct recognition of the inorganic constituents of the rubber, it being practicable to recognise the actual compounds that have been used, instead of relying on a fancy apportionment of acids and bases from the result of a complete but indiscriminating analysis. The determination of the organic matter left undissolved by treatment with petroleum may be effected when the analysis of the inorganic constituents has been made, by heating a known amount of the total insoluble matter with sulphuric acid, and weighing the mixed sulphates, provided that the previous analysis has shown that no metal forming an easily decomposable sulphate is present. Should the inorganic matter be completely soluble in acids, that fact can be taken advantage of instead of the former method being used. This plan is especially useful when graphite is present, as it frequently is. Rubbers containing surrogate of one sort or another, may prove difficult to dissolve in petroleum; but their case is provided for by the methods for the separation of surrogates (*v. i.*). Organic admixtures, such as fatty oils, paraffin and asphalt, are readily soluble in petroleum, while those diluents of which cork and wood refuse are a type, do not seem to be used in modern rubber manufacture, and only drag on a precarious existence in text books. With regard to surrogates, scarcely anything more than a qualitative recognition of their presence has been hitherto attempted, in spite of their great commercial importance. They consist chiefly of the products of heating fatty oils with sulphur, or their treatment with sulphur chloride, although blown oils that are free from sulphur are also used. Rubber surrogates are generally yellowish elastic substances, which frequently possess a moist and oily feel.

Two samples on analysis gave:—

	I	II.
Water	1.00%	0.85%
Sulphur	6.17	6.40
Ash	5.51	0.80

The ash consisted of lime and alumina, together with a little oxide of iron and silica. After several attempts at estimating surrogates in the presence of rubber, including the use of various organic solvents and the application of the Hübl method, the author arrived at the conclusion that alcoholic alkali was the best reagent for the purpose. Direct experiment showed that it was capable of dissolving surrogate completely, and that it had but little effect on vulcanised pure rubber, other than that of removing the superfluous sulphur. The chief drawback to its use is its tendency to leave a certain quantity of alkali in the undissolved rubber, which cannot be washed out, and thereby increases the weight of the insoluble portion. Digestion of the insoluble rubber with an acid is not advisable, as it is not perfectly effective, and much complicates the analysis of the inorganic portion. The phenomenon appears to be due to the formation of an alkaline salt with the rubber substance. The ash in the portion insoluble in alcoholic alkali is

not, however, a direct measure of the amount of alkali that has been taken up by the rubber, as a part of it consists of sulphur as sulphate, so that it is necessary to determine the quantity thus present and correct back from SO_3 to S. The number and kind of estimations required may be gathered from the following analysis.

PURE VULCANISED PARA RUBBER.		
Ash		2.80%
Total sulphur		9.50
Residue from extraction		95.55
Ash in residue (calc. on original rubber)		9.21
Sulphur in residue	"	4.35
Sulphur dissolved	"	5.15
Sulphur in ash	"	1.25
SO_3 equivalent to sulphur in ash (calc. on original rubber)		3.10
Na_2O in ash (by deducting original ash and SO_3), (calc. on original rubber)		3.31
Corrected residue from extraction		92.24
Soluble portion	Sulphur	5.15
	Organic matter	2.61

The extraction with alcoholic alkali is carried out by digesting 3 to 5 grams of the sample, cut into small pieces, with about ten times its weight of alcoholic soda, for 6 to 8 hours under a vertical condenser. The solution is turned out into a basin, diluted with water, which causes only a trifling precipitation, and evaporated until it no longer smells of alcohol, filtered through a weighed filter, washed, dried at 100°C . and weighed. The ash of the residue is determined on about 1 gram, ammonium nitrate being used to ensure the complete oxidation of the sulphur present. The portion of rubber substance that goes into solution may well consist of small amounts of vegetable fats that exist in commercially pure rubber, and possibly of a little unvulcanised rubber which has escaped the action of the sulphur, raw rubber being stated to be more readily attacked by alcoholic alkali than the vulcanised substance. The last hypothesis is, however, not borne out by direct experiment, as it was found that raw rubber yields about the same amount of soluble matter as does vulcanised.

The process is applicable to hard rubber, but the extraction takes place less readily. The results of analyses, on the lines given above, of rubber containing surrogate are quoted. The compositions deduced for two samples are as follows:—

	A.	B
Caoutchouc	41.32	84.43
Surrogate	51.68	8.31
Sulphur	5.10	4.26
Ash	1.90	3.00

What proportion of the sulphur belongs to the surrogate, and what to the vulcanised rubber cannot be determined by the method used. The analysis of hard rubber can, as mentioned above, be effected by the use of alcoholic alkali, but the determination of the inorganic constituents by means of petroleum is not feasible, for hard rubber, unlike soft rubber, dissolves imperfectly therein. By substituting paraffin wax at a temperature of 300° C., solution can be effected, though there is a risk of loss of sulphur, and a certain amount of carbonisation is apt to take place if the temperature be unduly high. The author is engaged in devising methods for the discrimination of other rubber surrogates, such as fatty oils, paraffin wax, and asphalt.

B. B.

The Distillation of Butyric Acid. H. D. Richmond (*Staz. Sper. Ag. Ital.* xxiii. 5).—The author disputes Duclaux's reasoning that the ratio between the amount of a solution of butyric acid which distils, and the acid contained therein, is expressed by a logarithmic formula; he gives the following formula $y = 2.22x - 0.0151x^2 + 0.000031x$ (where x = the amount of distillate from 100 c.c. and y = the quantity of butyric acid contained therein); from this he calculates that, were all the volatile acid in butter butyric, the quantity distilled when 110 c.c. are collected out of 140 c.c. should be 96.3 per cent., and when 50 c.c. are collected out of 75 c.c., 89.1 per cent. Actual experiments conducted as in the Reichert-Wollny process give 96.9 per cent and 89.6 per cent. respectively, using only a butyric acid solution, and 97.2 per cent. and 89.7 per cent. when 4.4 grammes and 2.2 grammes of well-washed butter acids are added; the insoluble acids, therefore, exert no retarding effect on the distillation.

The ratio between Reichert-Wollny figures (5 grammes of butter taken, 110 c.c. distilled from 140 c.c.) and Reichert figures (2.5 grammes of butter taken 50, c.c. distilled from 75 c.c.) should be by the equation 2.167.

Experimenting on butters he finds the following figures:—

Ratio	$\frac{R.W}{R}$	2.23
Percentage distilled	R.W	86.45
"	"	R 77.5

These figures differ notably from those required by theory; as the presence of volatile acids of higher molecular weight would cause the percentages to be higher than 96.3 per cent. and 89.1 per cent. and the ratio less than 2.167, he concludes that there is another volatile acid. He suggests the presence of lactic acid dissolved by the fat from the milk during churning.

H. D. R.

Some Analyses of "Cacio-Cavallo." G. Sartori. (*Staz. Sper. Ag. Ital.* xxii., 337).—"Cacio-cavallo" is the typical cheese of southern Italy. It is to some extent

made in Lombardy, but the chief centres of its production are Agro Romano, Albruzzi Puglie, Calabria, &c.

As the chemical composition of this important type of cheese has not yet been studied, the author considered it desirable to obtain samples from different sources; of the different samples obtained, only two prepared at the Royal Agricultural School of Scerni, were considered typical. Of these, No. 1 was made with whole milk, and No. 2, with a mixture of separated milk with sheep's milk containing 8 per cent. of fat; the first was pale-yellow, and had a normal odour; the second was deeply coloured, and had a slight smell of the sheep.

	No. 1	No. 2
Water... ..	19.756	22.090
Fat	36.706	35.900
Total proteids	37.825	36.063
Ash (without NaCl)	2.340	2.640
Sodium Chloride	3.260	3.164
Total	<u>99.887</u>	<u>99.857</u>
Pure Proteids	34.125	35.573
Ammoniacal Nitrogen0616	.0503
Amidic Nitrogen665	.609
Reichert-Wollny figure of the fat ...	25.30	28.71

The analyses were made according to the author's method for sheep's milk cheese (*Milch-Zeit.*, 1890, 51, *Annuario Lodi*, 1890); the pure proteids were separated according to Stutzer's method (*ANALYST* x. 75), with cupric hydroxide, and the nitrogen determined in the copper precipitate by Kjeldahl's method.

The author considers these results as only preliminary.

H. D. R.

The Analysis of Steam Lard. A. Goske. (*Chem. Zeit.*, xvi., 1892, 1560 and 1597).—The author has had experience in the analysis of steam lard in the works of A. Schöndorff & Cie., and the result of his observations is to show the futility of attaching the extreme importance to the indications afforded by the determination of the iodine absorption which is given to this criterion by some chemists. The usual assumption that a lard which gives the right iodine absorption must, if sophisticated, have been doctored by the addition of vegetable oil, and that consequently the qualitative disproof of its presence suffices with the determination of the iodine number, to place the sample beyond suspicion, is erroneous, because the proper consistency is not given to adulterated lard by the addition of vegetable oil, but by the incorporation of lard oil. (Contrast the results obtained in the examination of American lard; the *ANALYST*, Vol. xiv. 32.) The

following mixtures, all free from vegetable oil, give figures that approximate to that yielded by normal samples of lard, which is taken by the author as 60 :—

Mixture.	Per cent.	Iodine number (calculated.)
Pressed Beef Tallow ...	10	60·5
Steam Lard	90	
Pressed Beef Tallow ...	5	58·25
Steam Lard	85	
Mutton Tallow ...	30	57·5
Steam Lard	70	
Pressed Beef Tallow	25	59·75
Steam Lard	45	
Lard Oil	30	
Pressed Beef Tallow	35	57·27
Steam Lard	25	
Lard Oil	40	

In these mixtures the iodine number of pressed beef tallow is taken as 20, that of steam lard as 65, of mutton tallow as 40, and that of lard oil as 85. The corrective influence on the iodine number of the addition of lard oil to adulterated lard, of course increases the lower the temperature at which the oil has been pressed, and the more triolein it therefore contains. Thus a lard oil pressed at 34° C. had an iodine absorption of 70, and one pressed at 16° C. one of 82·8, numbers approaching those given by most of the vegetable oils suitable for use in sophistication.

The author having disposed of the claims of determinations, other than that of the iodine absorption to be considered certain criteria, proceeds to give the method of examination he adopts. The solidifying point is taken by gently melting the lard in a basin floating on water at a temperature of 50°-60° C., pouring it into a wide test tube up to a fixed height, surrounded by a bad conductor of heat, and observing the highest point attained in the act of solidifying as in Dalican's method, a thermometer divided to 0·2° C. being used. Some examples are quoted which show that butchers' lard (*Metzgerschmalz*) has a solidifying point of 28·6 to 29·9, and that pure steam lard ranges from 24·9 to 27·1, while the sophisticated lards vary from 29·8 to 36·6, there being a general tendency of the latter to be higher than genuine samples. A suspicious lard, with a solidifying point above 28° C. is then examined for the form of the crystals of "stearin" obtained from its solution in ether. 1 to 1·5 of a gram of a suspicious sample, or 2 grams of a sample that has the proper solidifying point, are taken, dissolved in 10 c.c. of ether in a test tube, the tube stoppered with a plug of cotton wool, and crystallisation allowed to proceed in a cool place for about six hours. As soon as a layer has formed at the bottom of the glass, but nowhere else, the supernatant liquor, which should still be quite clear, is poured off and replaced by a few c.c. of a colourless oil such as arachis or cotton seed, a

portion of the crystals picked out with a platinum loop, placed on a microscope slide and examined with a magnification of 300. The crystals first separating under these conditions are almost wholly beef stearin, and by their means as little as 5 per cent. of beef tallow can be detected. Mutton tallow does not crystallise so well, and 15 per cent. is the minimum quantity that can thus be recognised. The macroscopic aspect of the crystals is some guide to the adulterant present. Beef and mutton stearin form thick solid crusts, while lard stearin occurs in a looser state of aggregation. The microscopic appearances are equally definite. Beef stearin forms large tufts radiating from a common centre, the component needles being partly straight and partly curved. Lard stearin, on the other hand, though crystallising in tufts, is built up of plates instead of needles. The recognition of a vegetable oil is performed by the aid of the phospho-molybdate test, the nitric acid reaction and the rise of temperature with sulphuric acid. As beef tallow sometimes gives a blue or green colour with the first-mentioned reagent, like vegetable oils, it is necessary to test both the original lard and the oil expressed from it. Should the intensity of the colour be greater in the oil, it is clearly not due to the tallow. The same plan of separating the sample into a solid and a liquid portion, and testing both, is useful both for the nitric acid reaction and the rise of temperature with sulphuric acid.

B. B.

Analysis of Lard. C. Amthor and J. Zink. (*Zeits. f. Anal. Chem.*, 1892, xxxi., 534-537.)—In the following table the extreme and mean values of the numerous analyses of new and old samples of lard, tabulated by the authors, are given. The samples were rendered by the authors from different individuals and at different seasons.

			New.			Old.		
			Highest.	Lowest.	Mean.	Highest.	Lowest.	Mean.
Melting Point of Fat	49.0	47.0	47.8	48.0	47.5	47.7
Solidifying Point of Fat	29.5	27.0	28.2	29.5	29.0	29.2
Melting Point of Fatty Acids	45.0	42.5	44.1	47.5	45.0	46.2
Solidifying Point of Fatty Acids	39.5	37.0	38.3	42.5	40.0	40.9
Iodine Absorption of Fat	52.7	50.0	51.4	51.8	49.2	50.2
„ „ Fatty Acids	53.4	51.1	52.0	49.9	49.4	49.6
Acetyl Number	5.7	5.3	5.5	—	—	—
Saponification Equivalent	195.6	194.2	194.9	—	—	—
Hehner's Number	96.1	94.7	95.4	—	—	—

It is also stated that 0.0012 mgms. of KOH are required to neutralise the free fatty acids in 1 grm. of the fresh fat and 0.0015 to neutralise those in the same quantity of the old fat.

The age of the fatty acids decreases their iodine absorption very considerably and, as

might be expected, exposure to air does so to a greater extent. Thus, a sample of fresh fatty acids gave an iodine absorption of 51.61; after 42 days in a closed vessel this had fallen to 46.10, and in an open vessel to 44.34.

The authors have their say in the controversy as to the conditions under which the iodine absorption of a fat should be determined (compare ANALYST, 1892, 199). They conclude that, for lard at all events, an iodine solution which has been kept (45 days) according to Hubl's original method is preferable, and that the alleged necessity for a large excess of iodine does not exist; constant results were obtained whether the iodine was 21.2 per cent. or 84 per cent. in excess, or between these values. A. G. B.

The Determination of Lime in Basic Slag. A. F. Holleman. (*Chem. Zeit.*, xvi., 1892, 1471-1472.)—The conventional methods for the determination of lime in phosphates are not perfectly satisfactory when the phosphate is rich in iron and manganese, as in the case of basic slag. Both Classen's and Jones' process leave something to be desired, but an accurate method can be got by combining the two. The plan devised by the author is carried out as follows:—

50 c.c. of the acid solution of the slag, corresponding to 1 gram of the original substance, are evaporated to low bulk, 20 c.c. of a solution of neutral potassium oxalate (1:3) added, and digested on the water bath with vigorous stirring until the precipitate is pure white and free from clots, a result usually attained in about ten minutes. The precipitate is filtered and washed with hot water until oxalic acid is no longer recognisable in the filtrate. The filter paper is then perforated, the precipitate washed through, and the last particles dissolved off with hydrochloric acid (1:1), in which liquid the bulk of the calcium oxalate is also dissolved, about 15 c.c. of the strong acid being required for the purpose. The solution containing the whole of the lime is evaporated to 25 c.c., 10 c.c. of dilute sulphuric acid (1:5) and 150 c.c. of 96 per cent. alcohol added. The calcium sulphate is filtered after standing for three hours, washed with alcohol, and ignited and weighed in the ordinary manner. The test analysis on known mixtures and on basic slag are quite satisfactory, the results being appreciably better than those obtained by either of the constituent methods used singly. B. B.

THE ITALIAN SOCIETY OF ANALYTICAL CHEMISTS.

A society under the above title has been formed, and the inaugural meeting was held at Genoa on Aug. 27th, for the purpose of electing officers, framing bye-laws, and receiving scientific contributions.

The provisional committee consists of Drs. Musso of Turin, Longi of Rome, Leone of Palermo, Guelfi of Genoa, and Alessandri of Pavia. H. D. R.

REVIEWS.

TRAITE GENERAL d'ANALYSE DES BEURRES. By A. J. ZUNE. 108, Rue de Rennes, Paris, 2 vols., 25 francs.

Eighteen years have elapsed since the first method, based upon chemical principles for the analysis of butter fat, was published. The amount of literature which has meantime been given to the world on this subject is enormous, and Mons. A. J. Zune has undertaken a meritorious and arduous labour in collecting it, two volumes amounting to 800 pages being the result. The collection is most complete, the author having in the great majority of cases consulted the original papers. Perhaps he has gone rather too much into detail, even every insignificant contribution bearing upon the subject being noticed by him, the result being that he gives 9 distinct modifications of the process for estimating the insoluble fatty acids, and 14 for estimating the volatile fatty acids, each one being honoured by the author with the title of a distinct *procédé*. Thus we have the *procédés* Hehner-Angell, Lechartier, Perkins, Reichert, Meissl, Munier, Caldwell, Allen, Crampton, Schmitt, Wollny, Mansfeld, Nilson and Sendtner, for the estimation of volatile fatty acids alone. The differences are often ridiculously small, and such as would suggest themselves to any intelligent operator, but M. Zune discusses and criticises them all with the utmost gravity. As a collection of work done on the subject, the work is most valuable, the author showing himself an acute and intelligent critic of work done by others. The same can hardly be said of his own original efforts to devise a method of butter-analysis. The method proposed by him consists in the saponification of 0.5 gramme of butter-fat with baryta under pressure, removing the products of saponification soluble in water, namely glycerine and the salts of butyric, caproic, etc., acids, together with the excess of baryta by means of hot water, the caprate and caprylate by petroleum ether, the salts of the oleic series with pure ethylic ether, and those of the palmitic series by boiling alcohol, the equivalent being determined in each case. Every practised chemist will see that this cannot possibly be done with accuracy upon half-a-gram of fat; nor are the solubilities of the salts in the various solvents employed sufficiently satisfactory for good work. The author himself does not appear to have worked the process, for not a single statement of its results is given; while in other methods he is profuse in such statements. Many chapters are devoted to the examination of butter-fat by physical means. The microscopical examination is very fully discussed, and many well-drawn figures are given, showing the appearance of various fats when crystallized from solvents, etc.; but although the author strongly recommends microscopical examination, his own plates and drawings do not appear to the reviewer to furnish a distinctive means of discrimination. There is an immense amount of padding in the work, and more or less irrelevant matter; but this may be forgiven an author who has been evidently determined to collect everything touching the analysis of butter. In this task he has well succeeded.

O. H.

The Coal-Tar Colours : with special reference to their injurious qualities and the restriction of their use. A sanitary and medico-legal investigation, by Theodore Weyl, with a preface by Professor Sell. Translated by Henry Leffmann, M.B., Ph.D. Philadelphia ; P. Blakiston, Son & Co.

The existing information on the physiological effects of the coal-tar colours is largely the result of the investigations of the author, and chemists generally will welcome the appearance of the collected data in the English dress in which they are presented by Dr. Leffmann.

The little work in question is not restricted to a description of the poisonous characters of the synthetical colouring matters prepared from coal-tar, but deals also, in outline, with the methods employed for their manufacture and the chemical nature of the products. The laws in different countries relating to the use of poisonous colours, and the legal enactments concerning the use of such colours for the preparation of food, are fully set forth. Perusal of these sections shows forcibly the far more stringent regulations which exist in foreign countries, under which the colouring of "Demerara sugar" with a coal-tar dye, the nature of which was kept secret, would have been impossible. The injurious influence of the existing law of this country respecting experiments on animals is also brought into strong relief.

The typical reactions of the leading coal-tar colours are given at length, so that the analyst will usually be able to identify the substance with which he is dealing.

The book under notice deals with a number of important facts which have never received full recognition in any English work, and it will certainly be an indispensable addition to the library of the public analyst. The matter covers 150 pages, is well printed, and fairly free from errors. Dr. Leffmann deserves congratulation for his share in the production of a very useful volume.

A. H. A.

Olii Vegetali, Animali e Minerali (Vegetable, Animal and Mineral Oils). By G. Gorini. Second edition, by G. Fabris. Ulrico Hoepli, Milan, 1892. Price, 2 lire (1s. 8d.).

This little work is one of the well-known Hoepli Manuals of Science, Literature and Art. In this volume of 216 pages a large amount of information is given. It is essentially an elementary work, but most of the chief points in the chemistry and industry of oils are touched upon.

In the analytical part the leading characters, both physical and chemical, of the chief oils are given in tabulated form ; in some cases, notably in the table of refractive indices, the limits are too narrow, but generally the information is correct and up to

date. A considerable amount of space is devoted to the discussion of the electrical conductivity of oil, which is later declared to be a method of little practical use for analytical purposes.

We are glad to see that the presence of sulphur in oils from the *cruciferae* is not considered characteristic.

For determining the safety of mineral oils most importance is attached to the apparatus of Salleron and of Grainer ; no mention is made of the Abel tester.

It is, perhaps, too much to expect in a work of this order to find mention of all the latest researches, but we think that the work of Hazura and of Benedikt, on the chemistry of oils, and that of Salkowski, on the detection of raw oils in cod liver oil, should not have been ignored.

This volume should be of great use to students and beginners, and though it lacks a comprehensive index, is greatly superior to the ordinary class of "technical handbooks."

H. D. R.

CATALOGUE OF SCIENTIFIC APPARATUS AND REAGENTS. Brady & Martin, Newcastle-on-Tyne.

This is a fairly full and complete catalogue of Chemical and Bacteriological Apparatus, as well as of that appertaining to other Sciences. It is printed on good paper, the numerous engravings of apparatus are very clear and distinct, and the whole is enveloped in a neat binding. Full and useful explanations are appended to much of the apparatus. Altogether, it is superior to the ordinary run of such catalogues.

CORRESPONDENCE.

Mr. A. W. Stokes writes us as follows :—"In a paper by Mr. F. T. Shutt, published in the December number of THE ANALYST (vol. xvii., p. 227), on 'The Babcock Method of Milk Analysis,' he states—'Mr. Stokes quotes some of my results, neglecting others, and entirely ignores my conclusions.' In this he refers to my paper in THE ANALYST, vol. xvii., 127. Mr. Shutt is in error; I did not quote a single line of Mr. Shutt's paper from 'Chem. News, lxiv., 4.' I merely referred to the fact that Mr. Shutt does not find the Babcock method to give higher fats than reliable gravimetric processes. This reference occupied only two-and-a-half lines. The paper I wrote was not intended as a discussion on Mr. Shutt's paper. It is entirely different and altogether independent, as anyone may see by comparing the two."

APPOINTMENT.

The Town Council of the Borough of West Bromwich have elected Mr. John White, F.I.C., to the post of Public Analyst. Mr. White has been for several years chief Assistant to Dr. Bostock Hill.

THE ANALYST.

FEBRUARY, 1893.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE ANNUAL MEETING of the Society was held on Wednesday evening, January 4th, at the Chemical Society's Rooms, Burlington House, Piccadilly, Mr. Otto Hehner (the retiring President) in the chair.

The minutes of the last meeting having been read by the Secretary, and confirmed, the following gentlemen were elected as members :—Messrs. Arthur Frederick Morgan, F.C.S., analytical and consulting chemist, Newport, Mon. ; Cresacre George Moor, B.A., F.C.S., analytical chemist, London ; Thomas Shortbridge Tweedie, analytical chemist Annan ; and John Francis Liverseege, F.I.C., Birmingham, was elected an associate.

Messrs. W. C. Samuel, M. J. Sheridan, R. T. Thomson, and J. White were nominated as members.

In the absence of the Treasurer, through illness, Dr. Dyer presented the year's accounts. The balance-sheet was ordered to be printed and circulated in the usual way.

On the motion of the President, the Secretaries were requested to convey the thanks of the Society to the Chemical Society for the continued use of their rooms.

The retiring President then delivered the following Address :—

PRESIDENT'S ANNUAL ADDRESS.

GENTLEMEN,

It is a matter of some difficulty to compress within the short limits of the address which it is my privilege and pleasure to submit to you, an account of the many matters which have, during the past year, agitated the members of the Society and that section of the public which is interested in the working of the Sale of Food and Drugs Act. Never, since the passing of that Act, has it been subjected to so much comment and criticism as during the year 1892, and, indeed, many circumstances have combined to render it the object of discussion. We Public Analysts must welcome this attention which the Act, and, therefore, Public Analysts themselves, have received, in the full hope that, by any investigation or inquiry which may, and which, I hope, will be, instituted, some measure of recognition of the services of Public Analysts to the public will at length result.

We have to deplore the loss of four members by death during the past year, namely, of our most distinguished honorary member, Professor A. W. Hofmann, and three ordinary members, Dr. Theophilus Redwood, Mr. Norman Tate, and Dr. Tomkins. It is proper that I should further refer to the death of Dr. Albert Bernays, who, although not a member of our Society at the time of his death, was one of its founders and for years belonged to the Society. Almost up to the time of his death he was Public Analyst for several important metropolitan districts; and when the Society's Milk Committee went through its arduous labours, he most cheerfully and kindly assisted us.

It is not my intention on the present occasion to enlarge upon the loss which our profession and the whole world has sustained by the death of Dr. A. W. Hofmann. His name is a household word with every chemist, and will ever be written in the history of our science. I only desire to recall the fact that at one time he took a deep interest in the working of our Food Act. He was one of the commissioners appointed by the German Government to frame a Food Act for the German Empire, an Act which, though based upon our Act as a model, is in one important respect, to which I shall have to refer in the progress of this address, an improvement upon our own law, namely, by the institution of an imperial scientific authority, the Reichs-Gesundheits-Amt, in Berlin, which is the supreme advising authority in all matters concerning the Food Laws.

Dr. Theophilus Redwood, who died at a ripe old age, was the first president of the Society of Public Analysts, and shared with the late Mr. Wigner and the late Mr. Heisch, and with Dr. Muter, Dr. Dupre, and Mr. Allen, and a very few others, the arduous labour of collecting and of more or less binding together the heterogeneous elements which at first made up the Society of Public Analysts, and of doing whatever was possible to put the Food and Drugs Bill into the approximately workable shape which it ultimately assumed. The early history of the Society of Public Analysts was, as you all well know, full of troubles, and among these not the least was the resignation of Dr. Redwood. But after the period of the infancy of our Society had passed, and peace, as we happily enjoy it to-day, was established, we were all very glad to welcome Dr. Redwood back again as one of our members.

Mr. Norman Tate, of Liverpool, was for some years a vice-president of our Society. His work lay outside the ordinary occupation of the Public Analyst, but he always took a lively interest in our Society, and frequently attended our meetings; while those who came into contact with him more intimately will always remember the charm of his manner and his geniality.

Dr. H. Tomkins was Public Analyst and medical officer of health for the Borough of Leicester. He never practised as a physician, but devoted his life to sanitary science and preventive medicine. In private life Dr. Tomkins was kindly and amiable. He was not widely known; he chose rather to occupy his time entirely with subjects bearing upon his special work.

As to the general progress of the Society during 1892, we have every reason to be satisfied. We had at the time of the last annual meeting a total of 203 on our list, namely, 9 honorary and 172 ordinary members, and 23 associates. We have lost one honorary member, but the previous number of nine was made up by the election of our valued friend, Dr. P. Vieth, on his departure for Germany; while we have elected twenty new members, bringing the ordinary members up to 188. Four associates resigned or became members and we elected nine new ones, so that we now have twenty-five, or a total of all members of 222—nineteen more than last year. I must at the same time express my regret that there is every probability that several members will shortly be removed from our list on account of non-payment of their subscriptions, and I take this opportunity of begging the members to be a little more punctual in their payments, the secretarial work being materially increased by continual reminders which ought not to be necessary.

As regards our financial condition, you will have heard with gratification that, in spite of the heavy expenditure upon our Journal, we are yet some forty pounds better off than we were twelve months ago.

As regards the ANALYST, I hope all members appreciate the labour which has been bestowed upon it. It has been full of matter valuable to the Public Analyst and the Analytical Chemist and would have contained still more if the limited space of the paper had not necessitated the keeping out of a good many abstracts which, properly speaking, should have found a place in it. This difficulty will, in future, be to some extent removed by the increased size of the Journal, but the present increase is merely tentative, and is, I hope, only the precursor to still further enlargement. The enhanced price of the Journal will not, I think, be grudged by any subscriber, nor will any fault be found with the Publication Committee for deciding to materially advance the price of the back volumes, of which only very few are left. The labours of the sub-editor, Dr. W. J. Sykes, have often been considerable, and the Society owes him special thanks for the able and cheerful manner in which he has looked after the ANALYST. It appears very desirable to me that a general index should shortly be made of all the matter contained in the volumes of the ANALYST.

During the year, 43 papers were read at the meetings of the Society, and this is the largest number which has yet been recorded, being an advance of 18 upon the previous year. These papers were the work of 25 members. Thus one member out of every seven brought forward the result of some research, an indication of the great interest which is taken in the working of the Society. It seems, at last, as if the constant admonition of the successive Presidents to the younger members to come forward with a record of their experiences in the many branches of analytical work, was bearing fruit. I doubt whether any other scientific society of this country can boast of so satisfactory a record of work. Nine papers or notes were read by Mr. H. D. Richmond, four by myself, three by Mr. Stock, two each by Messrs. Allen, Dyer, Cassal, Heaton, Sykes, Leffmann and Smetham, and one each by Messrs. Embrey, Fisher, Gill, Leather, Lowe, Macfarlane, Moor,

Skertchly, Taylor, Teed, Stokes and Vieth. Although the year has not brought forth any paper of paramount importance, material advances have been made in several directions. In spite of this satisfactory record, I cannot help once more expressing the disappointment which I cannot but still feel that so many, in fact the majority, of members, never come before the Society with papers, although the observant analyst must almost daily come into contact with problems that require original research. I cannot think that there are any educated chemists, especially practical analysts, who do not daily recognise how much yet remains to be done and how little we really know, and who do not make honest efforts to throw here and there a little light into the dark corners which require exploration. It would be a useful work to point out, were it not obvious to every worker, in which directions useful labour might and should be bestowed. Even in a subject so widely thrashed out as that of milk, much remains to be done, since we cannot distinguish between milk naturally poor and that artificially let down by watering or skimming. I cannot help thinking that there *must* be differences, say, that the casein must be physically affected by extraneously added water. In butter, far more still is undone. Many ingenious but unsuccessful attempts have been made to elucidate its nature, and to show the differences between it and other fats. The differences established were sufficient when it was merely a question of differentiating between pure butter and unmixed other fat, but at the present time, where the addition of even five per cent. of margarine to butter pays the mixer very well indeed, the methods at our command are entirely insufficient. Yet none who has occupied himself with butter-research can doubt that there *are* essential differences between butter-fat and margarine, which differences should enable us to detect the addition of the smallest amount of the latter to butter. Absolute difference in constitution must be looked for, and not merely relative difference, as has been the case hitherto. In a matter apparently so simple as the estimation of chicory in coffee-mixtures, there is a field for useful and important research. It behoves us to point out at the present time, when legal limits are proposed to be fixed by Act of Parliament for coffee and chicory mixtures, that it is in the present state of knowledge quite impossible to say for certain within 5 per cent. more or less, or 10 per cent. altogether, how much chicory a certain mixture may contain. Both coffee and chicory vary far too much in composition, as regards almost every one of the constituents upon which the analyst usually relies, to allow of anything like an absolutely accurate determination. These differences were good enough while it was not a matter of very much consequence to arrive at the exact composition, and where no injustice was the result of a more or less erroneous report, so long as the fact of admixture of chicory was really proved beyond doubt. But when the analyst is called upon, as he may shortly be, to say whether a certain sample contains 49 per cent. of chicory or 51 per cent., the former percentage being legal, the latter illegal, it must be at once acknowledged that our knowledge fails. There can be but one way to get at these small differences, and that is, the absolute separation of the coffee from the chicory by mechanical means, and their

separate analysis. Similar remarks might be applied to our knowledge of cocoa, mustard, all the spices, and every natural article. Then there is the very large field of drugs, of the variation in chemical composition of which very little indeed is known from the point of view of the Public Analyst. Excepting a limited number of *chemical* drugs, as I may call them, the whole pharmacopœia is a dark continent to the chemist. There is hardly an extract or a tincture of the pharmacopœia that is analysable, except in the very crudest manner. I strongly hold that it is the duty of the State to see that officers appointed under one of its laws shall also be furnished with the means of carrying out their duties. Yet the authorities who composed that remarkable work, the "British Pharmacopœia," appear to have been utterly ignorant of the very articles which they admit into their pharmaceutical Bible, and the unfortunate analyst is expected to do that which the authors of the "Pharmacopœia" themselves have carefully avoided. Why, in the present state of knowledge, the analyst should be expected to pronounce definitely upon, say, a sample of compound tincture of rhubarb, or of lobelia, or of hops, or of pellitory, or of scores of others which may be sent to him by an Inspector under the Food and Drugs Act, or on any medicine or mixture of medicines, none of which may have an active or characteristic ingredient, or whose ingredients vary within wide and undefined limits, is more than I can comprehend. Yet such impossible work may lie within our province. If we had a Government fairly intelligently advised in such matters, such a Government would see that the analyst's duties should be practicable. The "Pharmacopœia" itself should by rights contain exact definitions, not only for the pharmacist, but for the chemist, of the articles it includes, and the State should give the means whereby they could be tested. It would be as absurd to send out an army without arms and boots and clothes, as to appoint the army of public analysts without giving them the weapons wherewith to fight the enemies of pure food and drugs. In Germany there is the Reichs-Gesundheits-Amt, that more or less supplies the necessary weapons, but here we are expected, for the beggarly fees which we receive, not only to do the routine work of analysing and giving an opinion based on the results, but also to work out our methods of analysis, no matter at what expenditure of labour and money, time and thought. And when satisfactory methods have been worked out, does a grateful country thank the zealous officer, who thus gives his brains and time, energy and money to the public without even asking for a recompense? No; we all know that every magistrate or sheriff sits in judgment over us, declaring, as happened quite lately in Scotland and also in England, that he knew as much about the merits of the lactometer in comparison with chemical analysis as the analyst himself. And do the Authorities which are set immediately above us—Somerset House and the Local Government Board—help in furnishing us with our fighting weapons? Again, we all know that this is not only not the case, but that every obstacle is placed in our way, and every means of discouragement adopted. And yet, I hope that Public Analysts in general and this Society in particular, will not swerve from the path they have adopted, and which has already, in spite of all, led to great and good results. There are remedies for the

present state of things, which I will venture later on in this address to suggest to you and which I hope will be taken into serious consideration by those in authority.

But, apart from purely public analyst work, untilled fields surround us and call for labour. For many years the chemist has been satisfied, in analysing animal and vegetable products, to split them up into broad groups of constituents; such as oil, that is, ether extract; albuminous matter, that is, nitrogen multiplied with a more or less arbitrary figure; acidity, that is, standard acid used multiplied with another arbitrary figure; and so on. Surely the time has come when such crude distinctions should be abandoned and an endeavour made to obtain definite scientific information. Each of the divisions of matters would afford a life-work to a number of chemists. But it would carry me altogether outside the limits which your patience and my time set me, were I to enlarge upon these almost untrodden fields. Once more I appeal to our members—and the Society is not only a Society of Public Analysts, but of Analytical Chemists—to see that they progress with the times. Routine chemists are not wanted; really scientific thought and labour are imperatively necessary. The time of bottle-washer-chemistry has gone by, never to return.

Your Council have had before them during the past year a number of matters of great importance to Public Analysts. We were threatened with a Bill to amend the Sale of Food and Drugs Act, introduced by Dr. Cameron, which, if it had passed without alteration, would have brought the Food Acts to a definite stop. It was proposed in the Bill that every invoice given by one trader to another should be considered a warranty under the Act. But inasmuch as the Act does not render the prosecution of the giver of the warranty or invoice-warranty compulsory, and no prosecution of a giver of any warranty has ever, so far as I know, taken place, every fraudulent vendor would have produced in court a paper exonerating him, and incriminating nobody. The Council interviewed Dr. Cameron, and recommended that Public Analysts should embody protests against the introduction of such a principle into his Bill, in their quarterly reports to their authorities, and a number of County Councils saw the danger and prepared themselves to take every step in their power against such a Bill passing into Law. I am glad to think that Dr. Cameron has since seen proper to amend his Bill in this direction. The Bill also contained clauses regulating the sale of mixtures of coffee and chicory, clauses which were quite unworkable, and the mischievous tendency of which was promptly pointed out by the President of the Local Government Board in an interview with representatives of the coffee trade.

During the year there took place some deliberations of a Departmental Committee of the Board of Agriculture, on the subject of passing an adulteration Act concerning manures and feeding stuffs. Several members of the Society gave evidence before a Committee of the House of Commons, with a view of preventing an Act analogous to the Food and Drugs Act being passed, and I am glad to think that the Committee's report is opposed to the Agricultural Adulteration Act. Extensions of the Food Acts

are certainly most desirable, as has been pointed out years ago by Mr. Allen and Mr. Cassal, but the whole Food Act requires revision before Public Analysts could consent to load themselves with additional duties on the same conditions as those imposed by the parent Act.

Representations have been made by the Council to the Local Government Board against the very unfortunate practice of some Local Authorities of combining the appointments of Medical Officer of Health and of Public Analyst in one. But very few Medical Officers have the chemical knowledge to become efficient Public Analysts. There are some brilliant exceptions, such as are afforded by the President-Elect, and by our Past-Presidents, Drs. Adams and Hill; but as a rule the average Medical Officer is not capable of doing the work of the Public Analyst in a manner creditable to himself or to the chemical or the medical professions. We Public Analysts have quite enough to do to keep up with the advances in chemical knowledge, and the Medical Officer has quite as much to do to keep abreast of Medical science. The Local Authorities who thus combine the appointments do so solely for reasons of so-called economy. They want to force the Medical Officer to do also the work of the Public Analyst without any, or without adequate, remuneration. One large town in the Midlands obtains analyses of seven or eight hundred samples per annum, representing a value of seven or eight hundred guineas, at the expense of the salary of an assistant to the Medical Officer of Health. This is intelligible from the "Vestry" point of view, but cannot be fair to the chemical or the medical professions. I have every reason to know that the best section of the medical profession is opposed to such a mischievous practice by Vestries and Councils, and would be very glad to see it put an end to. I am sorry to say that the correspondence which we have had with the Local Government Board has not so far led to a satisfactory result, but we shall continue our efforts in this direction. Ignorant Vestries and Boards may honestly think that a medical man may in cases be an efficient Public Analyst, but those of us who have had to do with the training of the medical student must know otherwise. In the end, no doubt, the separation of the two appointments will be obtained in every case. But it is essential that we, who are Public Analysts, shall show that we do represent a profession, that we are not mere amateurs, but life-long students, and that we will not do the work unless we are recognised as the medical profession *is* and *has* long been recognised. We may at present be paid like policemen or sanitary inspectors, but if each of us determines to be the authoritative adviser of his appointing authority without fear or favour, surely the time must come when we shall be listened to with as much respect as the medical officer is. Our work represents to the country the protection of monetary and other interests amounting to the greater part of the nation's earnings, and until we estimate ourselves at our proper value we must not wonder if Vestrymen and Councillors do not think highly of the services of the Public Analyst. Your council have felt that by the admission of unqualified men into the ranks of the Public Analysts the aim of the Society has been retarded, and they have given their serious attention to the

question of instituting a qualifying examination by which the knowledge of any candidate may be judged. In Canada, no analyst can be appointed until he has undergone examination before a special examining board appointed by the Governor in Council, and until he has obtained from such board a certificate setting forth that he is duly qualified to perform the duties attaching to the office of analyst (the Canadian Adulteration Act 48-49 Vict.c. 67). In England, the Vestry or the local Council is the judge of the qualification of the Analyst, and the Local Government Board confirms, I believe, in every case where no special objection is lodged and sustained. But in order to obtain a colourable qualification, intending candidates for the post of Public Analyst become members either of this or of the Chemical Society, although the membership of either at present in no way shows the qualification of the member. Although the deliberations of your council have not yet been carried to a conclusion, I am sure all qualified members of the Society will hope that ere long a scheme which will afford a means of showing the qualification of each Public Analyst will be properly worked out and enforced. It cannot be in anybody's interest to see inefficient men, or such as have gone through a few weeks' course of drilling in the laboratory of an Analyst, appointed to official and responsible positions; men who can perhaps mechanically make an analysis, so-called, of milk or water, but whose opinions are not worth having when given. Nothing but proper study, spread over years, can give the necessary knowledge and qualification. The Institute of Chemistry, which ought to be the proper qualifying body, has a Charter which does not admit of our objects being carried out under it, and, in my opinion, and in that of the majority of your Council, this Society owes it as a duty to the public and to its members to undertake the work to which I have referred. It must be remembered that the Analyst, once appointed, is the sole official chemical authority for a district, and the public have to go to him, whether he be competent or not, for advice and chemical help. Until, therefore, there is absolute security that the Analyst is really efficient, the confidence which ought to be reposed in him will never be generally established. I sincerely hope that we shall have the hearty support and co-operation of the Society in bringing about this much-needed reform.

For many years, indeed ever since the passing of the Food Act, the relations between Public Analysts and Somerset House have not been of that cordial character which, in the public interest, should obtain between officers working under the same Act of Parliament. The Somerset House Chemists undoubtedly fulfil one most useful function, namely, the correction of errors of analysis, which must, unavoidably, occur now and then, and against which there must necessarily be some authority to refer to. In the early days of the working of the Food Act, when experience was small, it was also well that some check should exist to prevent incorrect and unwarrantable conclusions being drawn from analytical data. It is to the credit of Dr. Bell and his colleagues that they have thus proved to be the regulators, as it were, of the analytical machine. Had it not been for the check thus imposed by Somerset House, our methods of analysis (especially of milk, which for years was analysed by a very bad and unscientific method of analysis) would

probably not have become what they are at the present time. But having said this, I regret that I cannot go further and credit Somerset House with any measure of cordial co-operation in our work. Upon Public Analysts alone has fallen the work of elaborating chemical methods, although, as I have taken occasion to point out before this evening, it is no function of theirs to supply the methods of analysis necessitated by their work. Upon them, also, has fallen the great labour of elaborating, upon the basis of an enormous number of analyses, the standards and limits from which to judge of the purity or otherwise of any articles submitted to them for analysis. They alone, not only unaided, but thwarted by Somerset House at every turn, have had to uphold satisfactory standards of quality for important articles of food, such as milk. When analysts of large experience find their views opposed by referees, doubtless acting from a strong sense of duty, but who have not one tithe of their experience; when they see that instead of what they believe to be broad and just views being taken, exceptional samples, which we all acknowledge to occur now and then, are ferretted out and held up as patterns whereby all other articles of the same class are to be judged; when they further see that conditions which are justly imposed upon them by the Act, referring to the state of the samples when analysed, are set at nought by the referees, it cannot well be wondered at that, with all desire to work in harmony for the public weal, they have not accustomed themselves to look upon Somerset House with any great amount of pleasure or enthusiasm. In the first years of the working of the Act, collisions between Public Analysts and the referees were frequent, not often resulting in favour of the correctness of the views of the latter; but gradually a settled state, of what I cannot describe otherwise than despair at a hopeless state of things, took hold of analysts, and they, against their better knowledge and belief, shaped their certificates in accordance with the views prevailing at Somerset House. Samples of milk and of butter were and are daily passed as genuine, which the analyst honestly believes to be adulterated, but which he cannot venture to condemn because he knows that, on reference to Somerset House, they would be pronounced to be genuine, or at least certified upon in ambiguous terms. The analyst came to the conclusion that it was not only useless to uphold his own views and to certify accordingly, but that it was injurious to the public interests to place himself in a position from which the authority of the referees might dislodge him and discredit the working of the whole Act. If, instead of milks being passed, as they daily are, with 2.5 per cent. of fat, or 8.3 per cent. of solids-not-fat, analysts, with the cordial support of the referees, had condemned them, the whole country would by this time be the gainer; bad breeds of cows would have vanished, injudicious feeding would have been given up, adulteration as a fine art would not have become possible, and every honest milk dealer would have rejoiced. If American States can pass laws laying down a minimum of twelve or twelve-and-a-half per cent. of total solids for milk as in Wisconsin, New Jersey, Minnesota; or, like Massachusetts, give the standard set by the "British Society of Public Analysts" the sanction of its law; or forbid, like Pennsylvania, the feeding of cows with distillery

waste, it appears very remarkable, to say the least, that we, the analysts of the country that passed the first anti-adulteration laws, should be debarred, by the action of Somerset House, from adhering to our own limits, limits which are followed by analysts over the rest of the civilised world. I am firmly persuaded that had it not been for the circumstances to which I have referred, we should long ago have done better, instead of worse, than the States to which I have referred. But, as I have stated, we resigned ourselves to what appeared a hard fate, until some months ago the Council of the Society once more made an effort to induce Dr. Bell to abandon the position which his department had taken up. We addressed a letter to him, which was signed by almost every Public Analyst of standing in this country, and by other members of the Society interested in the Sale of Food and Drugs Act, and which, therefore, may be taken to contain the almost unanimous expression of opinion of all British Public Analysts. We asked, in a respectful manner, that the form of certificate issued by Somerset House, which, while presumably intended to be unbiassed and judicial, had really a most harmful effect upon the magisterial benches to whom the certificates were addressed, should be abandoned, and that in doubtful cases Somerset House should state frankly that the results were compatible with the views taken by the Public Analyst; and that, in fact, a straightforward position should be taken up, without favour to anyone, either be it the trade or the analyst. We asked, further, that the practice of analysing milk which had, in consequence of keeping, undergone deep changes in composition, should be definitely abandoned. The public analyst is obliged, by the schedule appended to the Act of 1875, to say specifically whether the article, when analysed, had or had not undergone any changes that might interfere with the results of the analysis. Should the analyst state that a sample of milk analysed by him was decomposed, had fermented, or even become sour, would any bench in the kingdom condemn a vendor upon the analysis of such a sample? But Somerset House, though working under the same Act as ourselves, consider that they are not bound by the schedule, and habitually analyse milk which no Public Analyst who values his reputation would touch, much less certify upon. This was the essence of the letter which has been in the hands of each one of you. I much regret that Dr. Bell has not seen his way to accede to our requests, and that, as to the first point, he declines to alter the form of his certificate, and that, as regards decomposed milk, he alleges that he never analysed any milk which was, in his opinion, decomposed past satisfactory analysis. I need but state these facts, without much personal comment of mine, to bring you to the conviction that Dr. Bell has not well understood his duties to the public, or his obligations to his co-workers under the Act. We have cases on record in which the analysis of a milk when fresh, conclusively showed, upon the Somerset House limits themselves, that watering had taken place, but in which upon being analysed at Somerset House after a lapse of some weeks, was returned as genuine: the decomposition undergone by such samples had been smaller than usual, if one can speak of a "usual" amount of decomposition, and hence the constant allowance added to the analytical figures brought

up the solids-not-fat too high, and thus made the sample appear genuine. We have had at least one other case, in which the decomposition was greater than it ought to have been according to Dr. Bell—greater, I feel tempted to say, than the *official* amount of decomposition—the result being that the unwatered sample was declared to be watered by the referees. But it requires no cases and no argument to show how entirely fallacious any set time allowance must be, even if every pains are taken to estimate the amount of acidity and of alcohol produced during keeping. We cannot prescribe the number and kind of organisms which determine the extent and kind of decomposition, and the circumstances vary in every single case.

Yet we should not allow ourselves to fall into a state of hopeless despondency at such a sorry state of matters, for indications are not wanting that the public themselves are tired of the condition of things produced by the attitude of our referees on the one side and of 150 or more Public Analysts on the other. We would gladly bow to the decisions of chemists of acknowledged scientific authority; but when we believe, rightly or wrongly, that many Public Analysts have more experience and knowledge of the subject of food analysis than the referees, we are bound to make every effort to bring about an alteration. It would, of course, be far better, and in thorough harmony with our wishes, if such alteration could be effected by mutual consent; but if this be impossible, as I personally believe it to be, it is our duty to work for the best obtainable state of things, without regard for the sensibilities of individuals.

The defects of the Food Act, and the hardships which it entails to the retailer, have been largely discussed by the provision trade during the past year, and it must be very evident even to the casual observer, that changes must and will be made in the law as it now stands. No workable means have yet been proposed. There is much dissatisfaction among retailers, in that upon them falls all the burden and punishment for the sale of an adulterated article, although the offence may be, and in many cases is, entirely due to the wholesale house or to the manufacturer. In the case of proprietary articles, which are sold in packets, the contents of which cannot be interfered with by the retailer, there cannot be any good reason why the summons should not be made returnable against the manufacturer or the wholesale house instead of against the innocent retailer, as soon as it is proved that the latter sold these articles in the same state in which the wholesale house supplied them. But in the case of articles sold loose, which afford no proof of identity, such as milk, butter, cocoa, coffee, pepper, &c., the matter is far more difficult. It is hard upon the retailer to be summoned for an offence which he has not committed and against which he cannot well guard himself; on the other hand, the wholesale dealer cannot and will not take upon himself the responsibility for loose articles when they have once passed out of his hands, for he has no means of assuring himself that the retailer has not himself tampered with them, or substituted others for them. Between the retailer and the wholesale house any prosecution would be sure to fall to the ground if divided responsibility were accepted, as is now often urged by the trade.

If then, as it appears on all sides, an alteration in the law is demanded, namely, by the public, who urge that in many cases the law is a dead letter, and that there are no means of enforcing it; by the retailers, who justly complain that the law is all against them, and that it saddles them with responsibility which often should lie with others; by Public Analysts, who are handicapped in every way, hampered in their work, ignored by their appointing authorities and by many members of the bench; and by vestries and county councils, who when they do work the Act, often do so most unwillingly and fitfully, it may be pardoned me if, as your President, I also venture upon a number of suggestions, the outcome of a good deal of thought and experience, in which I have endeavoured to embody all that appears to me best of the many suggestions made by vendors and others interested in the matter. For, after all, the analyst, through whose hands go the samples of a number of counties, under the present Act representing widely varying circumstances—and who, at the same time, is often in intimate contact with the trade, and listens to the many suggestions and complaints made to him—must know better where the shoe pinches than the representative of any single interest or any single member of Parliament, who relies, perchance, upon the representations of some sanitary inspector. But, knowing that many interests are involved, I bring forward my proposals solely as suggestions, in the hope that some of them, at least, may be of use.

The want of uniformity among analysts and in the working of the Act is continually commented upon. This Society has done its best to induce its members to take up as uniform and reliable methods of analysis as the present state of analytical science allows, and it has, I think, fairly well succeeded in this. But there are hardly two authorities in the country who work the Act uniformly and consistently. Times out of number has the Local Government Board pointed out that, while in some cities or counties the utmost vigilance and even severity is shown, in very many others the Act is not worked at all, the officers being appointed with the full intention of not utilising them at all. The first thing necessary, therefore, is a central authority, which should not only be able to grumble, like the Local Government Board, but also to enforce. Whether this central authority be the Local Government Board or the Ministry of Agriculture matters not much, although a good deal is to be said in favour of the latter department of the Government, as adulteration affects agricultural interests far more than ordinary commercial ones. But I will assume that the Local Government Board, as hitherto, would be the guiding authority for the working of the Act. In order to enable that Department to give due weight to its recommendations it should, as in the case of medical officers of health pay part, say one-half, of the expenses of carrying out the Act, and receive an equal share of any fines which may be imposed or of other monies received. There should be a Chemical Officer of the Local Government Board, precisely as there are Medical Officers of the Department, who advise local authorities in all cases of difficulty, and who help and guide the Board and its officers. The chemical officer of the Board would also have the responsibility of any chemical matters that might come before the Board, such as water supply

sewage treatment, poisoning cases, &c., which matters are now, though largely chemical, left entirely in the hands of medical advisers. It is time that the Chemical Profession should be distinctly and not merely very indirectly represented in a Government Department. Such an officer would be solely an adviser, not a practising chemist. He would have to be a man of authority, whose word and knowledge could be relied upon, and whose work would be recognised by all sections of chemists throughout the country. He should not be chosen, as Somerset House has been chosen, solely for the sake of cheapness and convenience the Inland Revenue Laboratory being the only Government laboratory which existed at the time the Food Act was passed. The chemical officer should select a number of analysts and other chemists as assessors, chosen for their special knowledge in the various departments of chemistry coming before the Local Government Board, but who would not be permanent officers. This Chemical Board would have to issue in the form of regulations, alterable from time to time, standard methods of analysis to be followed by the Public Analyst, for all must have recognised by this time that figures obtained in food analysis depend largely upon the precise details of the method of analysis followed, and that all standards and limits which have been or may be fixed can be based solely upon definite modes of procedure. They would also have to lay down limits and standards for various articles, to be altered by regulation as any material advance is made in our knowledge. Other Governments have adopted this course. The Swedish and German Governments, for instance, not unfrequently issue regulations for the guidance of the analysts working under their laws, with precise and even the most minute details. No method of analysis and no limit should be embodied in any Act of Parliament, for Acts of Parliament are rigid and require years of agitation to get them altered, if the necessity for alteration arises. Any analyst devising a new and perhaps improved method of analysis would bring it before this Chemical Board in the full confidence that it would be fully and fairly considered, and without the fear of seeing printed in parliamentary blue-books reports totally at variance with the expressed opinions of the chemical advisers, as in a case within my personal knowledge.

This chemical officer, being in contact with all similar departments of Governments throughout the world, would be in a far better position to know what was going on in other countries than Public Analysts, and would be able to give early intelligence of new forms of adulterations springing up abroad. Had we had such a central guiding officer, the adulteration of lard with cotton oil, for instance, which flourished for some years before an English analyst found it out and brought it into general notice, would have been scotched much sooner than it actually was, for the American Government had the matter actually under consideration before we English analysts were aware even of the addition of cotton oil to lard.

If the Local Government Board paid part of the expenses of executing the Act we should get rid of two of the weakest points of the Act, namely the insufficient number of

samples taken by some authorities and the beggarly fees sometimes paid. The matter is not, let me plainly say, a money or fee question at all, but every labourer is worthy of his hire, and the Public Analyst having scientific and responsible work to perform, should not have to battle with some well-meaning, but intensely ignorant authority, consisting perhaps of dairy-farmers, who have heard that somebody or other analyses milk for a trumpery fee and forthwith expects the Public Analyst to do the same. Many Public Analysts, especially local men, with not too much of worldly goods and dependent largely upon their official work and what hangs around it, unwillingly give way, and the result is that the work is done at fees which are not sufficient to cover the salary of the laboratory boy, with the immense amount of responsibility thrown into the bargain. I cannot think that a professionally-advised Board would encourage this sort of thing. We should, secondly, get some uniformity in the number of samples analysed in various districts. Year after year the Local Government Board Reports show that certain towns and districts set the Act at defiance. In London, St. Martin's-in-the-Fields, in the country, Herefordshire, West Suffolk, Montgomery, and Radnorshire did not take any sample at all in 1891, nor did no less than 27 boroughs, all branded by the Board, without avail, as malefactors to their inhabitants. And there are quite a host of other towns where the number of samples falls altogether short of reasonable requirements. It is not too much to expect, surely, that one sample should be taken for every 1,000 inhabitants, as the Local Government Board recommends. To my thinking, a better mode of proportioning the work, would be to regulate the number of samples taken by the number of vendors of articles of food and drugs existing in each district. In towns the proportion of these is larger than in rural districts. Were there few vendors of food in any district it would be unreasonable to require many samples to be taken. From the census of 1881, the latest one available, I have compiled for some boroughs and counties in which I am specially interested the number of such vendors. Thus in Nottingham there is one vendor to every 41.11 inhabitants, in Nottinghamshire one to 47.96, in Derby (borough) one to 41.61, in Derbyshire one to 53.99, in Sussex one to 37.22. Assuming that to each establishment belong four persons on the average, I calculate that in Nottingham there are 1,136 establishments for the sale of food, drink and medicines, in Nottinghamshire 2,286, in Derby 488, in Derbyshire 1,789, and in Sussex 3,569. At the rate at which samples are at present collected and submitted to analysis in these districts a lifetime almost must elapse before each vendor is reached even once, that is to say, till one sample only be taken from each separate establishment. And when it is considered that each vendor deals in a number of articles, almost daily renewed, which are liable to adulteration, it should be obvious to the meanest understanding that the present working of the Food Act is utterly absurd and stupid, and that the small measure of moral influence produced is at least counteracted by the immense chance of non-detection. And if the Local Government Board cannot enforce its recommendations there is no hope whatever that the present state of things will be materially altered.

I would further put into the proposed Act, that the Public Analyst be chosen as hitherto by the local authorities and approved by the Local Government Board, with the understanding that only such men be appointed as have proved themselves to be qualified for the work. At present these things are managed by the very objectionable system of testimonials. I have seen testimonials given to candidates, by men who could not have the faintest knowledge of the duties of the Public Analyst; by professors, eminent in their departments, but not analysts; by medical authorities; by members of Parliament; and others. If the examination scheme, which I alluded to earlier in my address, were passed, as I hope it will be, and recognised by the chemical adviser of the Board, we should have the assurance that whatever the local influence of any candidate might be, (and local influence will always remain an important factor,) no inefficient man, no mere amateur, would be appointed. Even the most competent man *may* make a mistake, and where many samples are annually analysed there *will* always be a few mistakes, but no cases of gross incompetency—which give all opponents of the Act an excuse for crying out against the whole system of food examination—would occur.

Proper officers should be appointed whose duty would be the collection of samples. At present, police inspectors, well-known to every inhabitant of their districts, are largely employed. Police officers have their hands quite full enough with other work, and only undertake sample collection with great reluctance. Sometimes inspectors of nuisances or inspectors of weights and measures are the collecting officials, but these also, admirably as some of them perform their work, are full of other, and to them more important and congenial work. It would be as unreasonable to expect police officers to be inspectors of nuisances, or inspectors of weights and measures to perform detective work, as are the present requirements of otherwise occupied officials. The mode of collecting samples might otherwise remain the same. The division into three parts is just and fair, but the vendor should have the right of appending his own seal also, together with that of the inspector to each part of the sample. No single individual should have the responsibility thrust upon him of holding another man's reputation in his hands. Special regulations, binding upon all inspectors, should be issued by the chemical officer of the Board and his assessors for the proper putting up of the collected samples. At present samples are often secured in the most flimsy manner. I have had samples which could be readily removed from their packages without the breaking of any seal. I have seen coffee and mustard packed in papers which had soaked up much of the fat, so important to the analyst; spirits in wide-mouthed bottles with ill-fitting and porous corks, allowing of any amount of evaporation; I have had samples wrapped in arsenical paper, samples insufficient in quantity, like four ounces of beer, and monstrosities of all kinds. At present there is no means of regulating all this: each officer acts as he likes and sometimes resents any attempt at interference. There should, further, be special regulation, as to the division of samples like seidlitz powders, medicines, and aerated

beverages, which at present cannot be divided under the Act without rendering analysis impossible. The principle upon which the Act should be founded should be, that every purchaser should obtain the precise article for which he asks, unless he is distinctly and clearly informed at the time of the purchase, that the article demanded is not kept in stock. A mere label, therefore, should be no protection to the vendor if the label is not in accordance with the demands of the purchaser. Verbal notice should be given in every case, for some people may not be able to read, and others may not read the label at all. If, in my household, the servant is sent to the buttermilk man to get some butter, it is no use to me if the grocer sends margarine with a label intimating the fact. The servant does not bring the wrapper every time to me for my inspection. People may at first be offended if the grocer says that he has no tenpenny butter, but only margarine at the price; they may like to appear to be buying butter, when all the time they wish for margarine, but they would soon learn better, if they found that every trader gave them the same kind of notice. But if a label be appended to an article, I would hold the tradesman responsible for the accuracy of the description on the label. It is, I am told, the habit of some establishments to wrap all butter or margarine in margarine wrappers, to go quite safely under the present Act. I would punish a vendor for selling pure butter in a margarine wrapper, because the practice is plainly one intended to cover every fraud upon the ignorant and unwary. All mixtures should have on the label the proportion of the ingredients, excepting in the case of proprietary preparations, which may be composed of substances or made up in proportions which it might not be fair to require to have stated. But in these cases I would let the vendor omit to state the constituents and their proportions at his peril and on his responsibility. Every third portion of each sample taken, should, as at present, be retained by the inspector. In the case of milk, the only article so perishable that, if any check analysis is to be made, it must be made quickly and before decomposition has set in, I would make it incumbent upon the analyst to report within one week, or even within four days of the receipt of the sample, a report to be sent both to the inspector and to the chemical officer of the Local Government Board if it is found to be adulterated. In that case the inspector would immediately forward his portion of the sample to the Chemical Officer of the Board, by whom it would be preserved by heat sterilisation, or be opened in order to receive a proper amount of some preservative like chloroform, carbon disulphide, potassium bichromate, or other suitable antiseptic in exactly ascertained quantity, and there again sealed up for reference if required, should a dispute arise. In that case the sample would be sent to one of the Chemical Officer's assessors, a man specially experienced in milk analysis, and who could speak on the matter with authority, to be analysed by him; provided always that the assessor should not be interested in any way in the particular case. If, therefore, the assessor were a Public Analyst, as would be probable, no samples from his own districts could be sent to him for reference, and therefore for each article two assessors would have to be appointed, one as principal, the other as deputy in cases such as that referred to.

If a dispute arose in any other article, the sample would also be sent to the Chemical Officer, and would be placed by him in the hands of another suitable referee. In this way each article would be judged under the recognised rules of the department as to method of analysis and standard adopted, by men specially experienced in each special article. I would, for instance, rather have a drug referred to Dr. Muter or Mr. Davies, a sample of wine to Dr. Dupre, one of oil to Mr. Allen, one of milk to Dr. Vieth, Mr. Richmond, or Mr. Faber, than any of these to the present referees without special experience in any single department. Thus we should once and for all get rid of unseemly disputes, and the trades as well as the analyst would have due protection and the best advice obtainable. The summons should in every case be returned against the actual vendor, who, if he alleged that he did not commit the offence or acted in ignorance, might be required by the Act to state this to the Inspector serving the summons, whereupon for the vendor's protection the Inspector would take a sample of the same article next delivered to the retailer by the wholesale house, before it was touched by the retailer, obtain an analysis which, if it proved unfavourable, would exonerate the retailer, but would incriminate the wholesale dealer, while in the contrary case the retailer would be summoned for the former sample and punished. As the wholesale house would be quite ignorant of the result of the first analysis, they would have no chance, should they really be the guilty parties as alleged by the retailer, of sending a specially pure article, and the retailer would not only have no interest, to put the wholesale dealer upon his guard, but the contrary, as he would make the punishment come upon himself. In this manner, I think, the retailer as well as the manufacturer would be duly protected, at the least possible inconvenience to himself or cost to the authorities. No firm given to adulterating their goods does so only occasionally, but rather as a matter of principle and daily, in the hope, which is at present but too often realised, that the fraud will not be detected, seeing that the Inspector can come but very rarely indeed. On the other hand, mistakes which may occur in the best-regulated businesses, such as the accidental substitution of margarine for butter, would be more or less distinguished from intentional fraud.

I know, that in many minds there is an objection against any form of centralisation, and justly so, but, having regard to the working of the decentralised Food and Drugs Act during the last seventeen years some such plan of semi-centralisation appears to me to be the only solution of a remarkably difficult problem. Nothing certainly could be worse than the present system of making every individual analyst the judge of what ought and what ought not to be allowed. There are sometimes reasons, which cannot be known to the analyst, why certain admixtures should be made to food or drink, which can now only be brought forward at the expense of a law case, involving the reputation of honest firms, or the good name and repute of the analyst. Such cases would come first under the notice of the Chemical Board, and due allowance would be more likely to be made for trade requirements than is the case at present.

I fear I have tried your patience to the utmost extent, but the importance of the subject to many outside this Society, as well as to its members, must be my excuse.

I now take leave of you as your President. I thank you one and all for the unvarying courtesy and friendship shown me during the two years I have had the honour of presiding here, two years which leave nothing but pleasant memories with me. It has been a great pleasure and satisfaction to me, who have worked for so many years as an active member, to see the Society increase in numbers and in standing, and I hope I have to some measure, at least, justified the promise I made two years ago, that the interests of the Society would be watched over by me jealously and to the utmost of my power.

Sir Charles A. Cameron, in proposing a vote of thanks to the President for his address, said there was not a single sentence in the valuable address to which they had listened—except that relating to the proposal to institute diplomas in connection with the Society—which would not have the cordial assent of every member. Perhaps that was the only vexed question the President had touched upon, but he thought they would all heartily agree with the other proposals in the address. He thought Mr. Hehner had shown them all the difficulties under which Public Analysts laboured, and that he had thrown out a number of valuable suggestions which he (Sir Charles) hoped would be incorporated in whatever future Act of Parliament might be passed in reference to the adulteration of food and drugs. He thought the thanks of the Society were due to the President, not only for his address, but also for the great zeal and interest he had exhibited in the welfare of the Society during the two years he had had the honour of being its President. He (Sir Charles) regretted that he could not often be present at the meetings of the Society, and therefore could only repeat what he had gathered from his friends who were members of the Society, as to the close attention Mr. Hehner had given to his functions during the last two years. From every quarter he had heard that their President had been unremitting in attending to the duties of his office, being invariably present at all the meetings of the Society, and the meetings of the Council and sub-Committees. He therefore ventured to say that they would remember for many years the presidency of Mr. Hehner, because, not only had he been an admirable President from a purely scientific point of view, but his pleasant and practical method of discharging the duties of the chair was such as to commend him to their gratitude and thanks. His address had not only been a very suggestive one, but the numerous references he had made to certain points had caused them, notwithstanding the low temperature of the room in which they were assembled, to “warm-up” a little. He had great pleasure in proposing that the heartiest thanks of the Society be tendered to Mr. Hehner for the admirable way in which he had discharged the duties of President during the last two years, and for the valuable and suggestive address he had delivered that evening.

Mr. Alfred H. Allen, in seconding the vote of thanks, endorsed the complimentary references which Sir Charles had made to Mr. Hehner's services as President, and in alluding to one or two points raised in the address, suggested that that was not the occasion for discussing them, as their time that evening was limited. He thought it would be profitable if the Society devoted its next meeting, even if they did not have a special meeting, to discussing the address further. They could then have the propositions in print and would be able to consider them in detail. Certainly they should not be allowed to drop. The vote was then put to the meeting and carried by acclamation.

The President, in acknowledging the compliment, referred to the diploma question. He said he knew the subject bristled with difficulties; the more they looked into it the more hopeless it seemed to be to satisfy everybody. He thanked Mr. Allen for his suggestion, though he would not take it as an indication that anything he (the President) had said would necessarily be received with approval. He cordially welcomed the suggestion in view of the immense importance which the subject was likely to have in the future. It would be a misfortune if members of the Society came with conflicting views before a Parliamentary Committee.

The next business before the meeting was the election of the Officers and Council for the new year, and, the nominations submitted to the members having been opened and examined by the scrutineers, the following gentlemen were declared elected:—

President—Sir Chas. Cameron, M.D., F.R.C.S.

Vice-Presidents (who have filled the office of President)—M. A. Adams, F.R.C.S.; A. H. Allen; A. Dupré, Ph.D., F.R.S.; Otto Hehner; Alfred Hill, M.D.; J. Muter, Ph.D., M.A., F.R.S.E. (Who have not filled the office of President)—Charles E. Cassal; Sidney Harvey; Thomas Stevenson, M.D., F.R.C.P.

Treasurer—C. W. Heaton.

Hon. Secretaries—R. H. Davies, Bernard Dyer, D.Sc.

Other Members of Council—C. M. Aikman, M.A., B.Sc., F.R.S.E.; R. Bodmer; William Chattaway; John Hughes; E. W. T. Jones; H. Droop Richmond; Alfred Smetham.

The names of those Members of Council whose term of office has not yet expired, and who consequently do not retire this year, are T. Fairley; W. J. Sykes; F. L. Teed; E. W. Voelcker; and C. R. A. Wright.

Mr. Hehner formally retired from the chair in favour of the newly-elected president, Sir Charles Cameron.

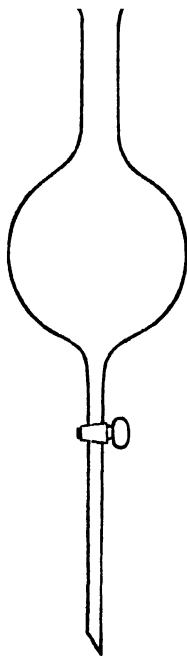
Sir Charles Cameron acknowledged the honour conferred upon him in suitable terms, and, in alluding to the regularity with which his predecessor had attended to his duties, pointed out that his (Sir Charles') residence beyond the Irish Sea precluded him from hoping to follow his predecessor's example in that respect so well as

he should like. He was sure, however, they had fully considered the circumstance when they asked him to accept the post of president, and he could only attribute the kindness they had thus shown him not merely as a compliment to himself, but to the members of the Society resident in Ireland. He could only say that he very highly appreciated the compliment they had paid him in selecting him as their President, especially as the number of members of the Society resident in Ireland was not large. He should do the utmost that was in his power to attend the meetings of the Society, and he might tell them this much, that if ever a channel tunnel were established between Holyhead and Kingstown, or, as it had been proposed, between the north of Ireland and Scotland, he was such a bad sailor that he should always prefer to either ride across the channel through a tunnel or across it by some organised system of ballooning. He should then be one of the most punctual members of their Society, but, as he was afraid next year would not see any such improved means of communication between the two countries, he should content himself by looking at the weather charts throughout the year, and when a period of calm coincided with a meeting of the Society, they might be sure he would turn up.

The following paper was then read :—

THE ESTIMATION OF INSOLUBLE FATTY ACIDS.

BY CHARLES E. CASSAL.



THE use of a filter in the process of washing insoluble fatty acids in the analysis of butter and other fats is cumbrous and presents numerous and obvious sources of error. In carrying out this estimation I have for several years employed a form of separator which renders the use of a filter paper quite unnecessary, and at the same time enables the washing to be done in a far more thorough and satisfactory manner. In the form in which it was originally used, the separator was a pear-shaped vessel of rather thick glass, provided with a tapped tube with a narrow bore; but the apparatus that I have used for some years past is globular, is made of thin glass, will stand boiling water, and is provided with a tap and tube of wider bore, the end of the tube being cut off at an angle. The older form was more liable to break, and did not always allow of the fatty acids being obtained in a thin layer, which is, in many respects, a great advantage; nor of the greater part of the acids being easily removed from the apparatus without the aid of a solvent.

In using the apparatus, the soap obtained from any convenient weight of fat, but preferably from about five grammes, is poured into strong hydrochloric acid previously introduced into the separator, the residual soap being washed in through a funnel with boiling water.

The resulting fatty acids are washed with successive small quantities of boiling water, the globe of the separator being about half-filled each time. After the decomposition it is necessary to allow the mixture to stand; the major portion of the insoluble acids appear, of course, as an oily layer on the surface of the liquid, but a very marked proportion is distributed through the latter and makes it more or less turbid. Hence it is convenient so to time the work as to allow of the first mixture standing over night. This always ensures complete separation, and subsequent washing does not reproduce the turbidity. The washing being completed, the last wash-water is run off, and the insoluble fatty acids, which are, of course, in a liquid state, are received directly into a weighed vessel, preferably a deep and light platinum basin. The portions adhering to the sides of the apparatus are then washed down with ether, the latter evaporated, and the acids dried to constant weight. The apparatus will be found useful for several other operations.

Three other papers were postponed owing to the lateness of the hour.

The meeting then adjourned to the Criterion Restaurant, where the Annual Dinner of the Society was held. About fifty members and friends assembled. The chair was occupied by the newly-elected President, Sir Charles Cameron, supported by—Mr. Otto Hehner (past President), Dr. James Bell, C.B., F.R.S., Captain Verney Cameron C.B., R.N., Dr. F. Allan, Surgeon-Major Carte, Dr. Cory, Dr. Abraham, Surgeon-Captain Jackson, Dr. Kennedy, Mr. A. H. Allen, Mr. C. E. Cassal, Dr. Sykes, Mr. E. W. Voelcker, Mr. R. H. Davies (Hon. Sec.), Dr. Bernard Dyer (Hon. Sec.), Mr. Aubrey Rake (Solicitor to the Society), &c.

The Chairman proposed the toast of the Queen, the Prince and Princess of Wales, and the rest of the Royal Family; and that of the Navy, Army and Reserve Forces, the former being responded to by Captain Cameron. Surgeon-Major Carte replied for the Army, and Captain Cassal for the Reserve Forces.

The Chairman proposed the toast of the evening: "The Society of Public Analysts," coupled with the name of Mr. Otto Hehner, ex-President, to whom he referred in very flattering terms.

Mr. Hehner said the Society had for seventeen years worked very hard for the welfare of the community under circumstances which would have disheartened most other people. When, twenty-one years ago, the Adulteration Act of 1872 was passed, and the fiat went forth that there should be a body of gentlemen appointed as Public Analysts, there were no practical means of carrying out the requirements of the Act, and although they were asked to make analyses of foods and drinks they had practically no means of doing so. They knew now that twenty-one years ago they were intensely ignorant, and knew nothing of such matters as milk, butter, bread, and drugs; but the Society had laboured year after year in connection with these subjects, and members from all corners of the country had read a great many papers. They were, he thought, in a far

better position now than they were before. In the seventeen years the Society had existed its members had read 450 papers on every possible subject of their work, and, besides that, 150 papers had been published by members in the Society's Journal. This constituted a mine of information for all those who worked in that direction, and especially for Analysts all over the world, for this country was the first to take action in the matter, and the Legislatures of other countries had followed its lead. Seventeen years ago 20 per cent. of the articles analysed were adulterated, now the percentage was about 11. As to the quantity of adulteration then practised, it was appalling—milk would be found to have 50 per cent. of added water; bread had any amount of alum in it; and spirits were watered to any extent. Now, however, there was a general decline in adulteration, and thus the Public Analyst had saved the public millions of pounds, and inasmuch as the larger portion of the Nation's earnings were spent in food, the importance of their work was manifest. But as the Analysts had made advances in knowledge, so had the enemies of the purity of food, and it must be remembered that while the former worked in the light, the latter worked in secret, and had taken such advantage of the increase of knowledge, that at the present time, more than at any other time in the history of the world, adulteration was practised as a fine art. There was, therefore, still plenty of work for Public Analysts to do in coping with scientific adulteration.

Mr. Alfred H. Allen proposed the next toast, the health of the President, Sir Charles Cameron, who, he said, was the senior Public Analyst of the United Kingdom, he having been appointed under the Act of 1860, which was then only a Permissive Act. They might fairly say that Sir Charles had a longer experience of the working of the Adulteration Act than any Analyst now living, and they had, therefore, every reason to regard him as their father so far as experience went in these matters, and as such they cordially welcomed him to the Presidential Chair.

The President in response, referred to his experience as a Public Analyst during the last thirty-one years, and contrasted the conditions under which he had to work during the first part of that period with the altered circumstances of recent years. There was then very little to guide Public Analysts, and there was no awful tribunal at Somerset House to overrule them. So far as Dublin was concerned at the present time, there was hardly anything in the way of ordinary articles of food adulterated, with the exception of butter and milk. With regard to flour, bread, coffee, confectionery, etc., they were formerly adulterated to an enormous extent, but it would be impossible now, he thought, to find any of these things in a sophisticated state in his city. In concluding, Sir Charles referred with satisfaction to the honour that had been done the country he represented, by the fact that quite recently he had not only been appointed President of that Society, but also President of the Sanitary Institute of Great Britain and Ireland, and also of the Institute of Public Health, of which his friends, Dr. Allan and Professor Smith were officers.

Mr. Newlands proposed, in felicitous terms, the health of the Vice-Presidents, the Honorary Secretaries, and the Solicitor, coupling with the former the names of Dr. Adam^s and Mr. Cassal. The latter in reply spoke of the vast strides made by the analytical profession during the last few years, and said he thought they would look forward with confidence to the time when every member practising in the profession would be regarded as a valuable and honoured servant of the country.

Dr. Bernard Dyer, in responding to the toast of the Hon. Secretaries, referred to the work of the Society during the last nine years, and the labours of his predecessor, the late Mr. Wigner. He referred in cordial terms to the pleasant and harmonious way in which his friend Mr. Davies had, since Mr. Hehner's elevation to the chair, collaborated with him in the Secretarial work. He was sure they all regretted sincerely that Dr. Muter, who had served the Society as Vice-President for many years, and who was a familiar figure at their festive gatherings, was too unwell to be present on that occasion. He had received a letter from their honorary member and Past Vice-President, Dr. Vieth, who was at present resident in Germany, referring to the many happy hours he had spent with the members of the Society when attending their meetings in England, and the pleasure he experienced in receiving "THE ANALYST" from time to time, that being the only paper he had read with interest since he had left the country.

Mr. Davies and Mr. Aubrey Rake having also suitably responded.

The Chairman proposed the toast of the "Guests," coupled with the name of Dr. James Bell.

Dr. Bell, in replying to the toast of the "Guests," said with regard to the President's cordial extension of the invitation to future dinners of the Society, he would be glad to respond at once and affirmatively to that invitation. He had felt a special interest in attending this dinner, for he naturally had very strong sympathies with professional chemists. He knew from experience the anxieties and difficulties they had to contend with, because those anxieties and difficulties corresponded with his own, and they were often great. He hoped that in the not distant future Public Analysts might be placed in a more satisfactory position than they at present occupied in the discharge of their local duties. Local authorities were often bodies whom it was almost impossible to satisfy, and he felt strongly that Public Analysts should be placed in a more independent position. The Society of Public Analysts had a splendid record. It had done a mass of excellent work, embodied in a series of admirable papers; it had devised many excellent processes of analysis, and it deserved the sincere thanks of the chemical profession generally. He wished it to be understood with regard to his official position that he was no conservative. He had followed every important process and investigated every promising method that had been brought forward under the auspices of the Society, and he had never hesitated, as soon as he was satisfied of their usefulness and their accuracy, to adopt such processes. His official position undoubtedly was sometimes a painful one.

He regretted most deeply whenever he was obliged in any point to go against any Public Analyst in a matter of reference. He was glad to say that the great improvements in analytical work had, during recent years, relieved him of an important part of his own anxieties. Out of all the cases referred to him under the Sale of Food and Drugs Act during the last nine months, he was pleased to be able to say that there were only three cases in which he had felt himself obliged to differ from the opinions expressed by the Public Analysts. This, he thought, was extremely satisfactory and he believed that errors were now reduced almost to a minimum. He sincerely congratulated the Society on its progress and hoped it would continue, and as time went on he trusted that they would never have occasion to be apprehensive of the result of references to Somerset House. (Applause).

Dr. W. R. Smith proposed "THE ANALYST," and associated with it the name of its hard-working and energetic sub-editor, Dr. W. J. Sykes.

Dr. Sykes briefly returned thanks, and the proceedings shortly afterwards closed.

The pleasure of the evening was greatly enhanced by selections of vocal music contributed by Surgeon-Major Carte, Dr. Abraham, Mr. W. Bateman, Captain Watson, and other guests and members.

A special meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on January 18th, for the discussion of the retiring President's (Mr. Otto Hehner's) Annual Address.

The Assay of Crude Carbohc Acid. G. Schacherl. (*Zeits. oesterr. Apoth. Ver.*, 1892, xxx. 794, through *Chem. Zeit.*)—The usual method of shaking out the phenols with caustic soda is liable to give inaccurate results, as a certain amount of resinous constituents and some neutral tar oil are also dissolved. The plan advocated is as follows:—100 c.c. of the crude carbohc acid are taken if the sample be of poor quality, and 50 c.c. in the contrary case. The measured portion is then placed in a separating funnel and shaken out with 100 c.c. of caustic soda of sp. gr. 1.1, the alkaline liquid separated, and the shaking repeated two or three times with further quantities of 50 c.c. The separated liquid from the last extraction should give no oily drops when acidulated. The combined alkaline extract is then diluted with an equal volume of water, and distilled in a flask of about one litre capacity. When the distillate comes over free from oily drops, the removal of the neutral oils may be considered complete. The contents of the distilling flask are then allowed to cool, acidulated with hydrochloric acid and distilled again, using a large condenser. When the distillate, consisting of phenols accompanied by water, amounts to about 200 c.c., the operation is stopped, and the water separated from the phenols and returned to the distilling flask. The distillation is proceeded with and the collection of the phenols effected as before, the treatment being repeated until the whole of the phenols have come over. The last distillate, which should measure about 60 to 70 c.c., is supersaturated with salt, and the separated phenols read off and their volume added to that of the main quantity.

B. B.

THE ANALYST.

MARCH, 1893.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

A SPECIAL Meeting of the Society of Public Analysts was held in the rooms of the Chemical Society, Burlington House, on February 15th, 1893. The meeting was convened to consider a series of resolutions drafted by the Council on the subject of certain proposed amendments in the laws relating to adulteration. There was a large attendance of Members of the Society, and of other gentlemen interested in the working of The Food and Drugs Acts. In the unavoidable absence of the President (Sir Charles Cameron), the chair was taken by Mr. M. A. Adams. Amongst those present were Dr. Cameron, M.P., The Hon. H. A. de Tatton Egerton, M.P., Mr. Kearley, M.P., Mr. G. M. Allender, &c. Letters of regret at inability to be present were read from Sir C. Cameron, President of the Society, Sir Walter Foster, M.P., Mr. Brunner, M.P., Col. Howard Vincent, M.P., Mr. H. J. Wilson, M.P., Sir Henry Thompson, Sir W. Pink, Sir Joseph Fayrer, and many others, including most of such provincial Public Analysts as were unable to be present. The following resolutions were put to the meeting and carried unanimously:—

1. That amendment of the laws relating to adulteration is urgently required.
2. That the present Acts often operate unfairly on the retail traders, and that provision is necessary to ensure in many cases of adulteration the prosecution of the real offenders.
3. That in view of the fact that, as is shown in the Local Government Board reports, the Food Acts are practically dead letters in a large area of the United Kingdom, it is necessary that adequate provision be made for securing uniformity in their application and in their due enforcement.
4. That in order that the Local Government Board should have better control over the working of the Acts, a portion of the expenses of working them should be borne by the Imperial Revenue.
5. That in view of this, it is desirable that there should be a duly constituted Chemical Department of the Local Government Board with whom the Public Analysts, as officers of the Local Government Board, should be placed in direct relation.

6. That the present system of reference in the case of disputed analyses is unsatisfactory, and ought to be entirely remodelled.
7. That the compulsory combinations of the two offices of Medical Officer and Public Analyst is in the public interest undesirable.
8. That provision should be made to ensure better than heretofore the proper qualification of officers under the Act.

A full report of the meeting will appear in our next issue.

The usual Monthly Meeting of the Society was held on Wednesday, 1st February. In the absence of the President, on the motion of Mr. Hehner, seconded by Mr. John Hughes, the chair was taken by Mr. Alfred H. Allen.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed as members:—Mr. Thomas Richard Duggan, F.I.C., F.C.S., Sunny Bank, Vanbrugh Hill, Blackheath, S.E.; Mr. W. H. Symons, F.I.C., F.R.M.S., F.C.S., 130, Fellows Road, South Hampstead. Mr. W. S. Crocker, assistant to Mr. J. Brierley, and Mr. James Sykes, assistant to Mr. G. Jarman, were proposed as associates.

Messrs. W. Cobden Samuel, M. J. Sheridan, B. T. Thomson, and John White, were duly elected members of the Society.

Mr. Richmond then read the following paper:—

THE COMPOSITION OF MILK AND MILK-PRODUCTS.

By H. DROOP RICHMOND.

THIS paper is a continuation of the annual reports of Dr. Vieth on his work in the laboratory of the Aylesbury Dairy Company. The work done in 1892 is treated of in the present communication. (For previous reports, see ANALYST, vii. 53; viii. 38; ix. 56; x. 67; xi. 66; xii. 39; xiii. 46; xiv. 69; xv. 44; xvi. 61; and xvii. 62).

The total number of samples analysed in 1892 was 25,931, viz.:—

23,865 samples of milk.		
1,338	„	cream.
566	„	skim-milk.
8	„	butter-milk.
78	„	butter.
24	„	water.
22	„	sundries.

Of the milk samples 13,196 were taken from the delivery churns on arrival of the milk from the country. The bulk is distributed with the least possible delay to the

customers, portions being, however, kept for the production of cream. For the control over the men employed in delivering the milk, a further 9,103 samples were taken before, during, and after delivery, and analysed comparatively. The following table contains the result of these analyses :—

	On Arrival.				Before Delivery.	At Commence- ment of Delivery.	During Delivery.	After Delivery
	Spec. Grav.	Tot. Sol.	Fat.	S.-N.-F.	T. S.	T. S.	T. S.	T. S.
January ...	1·0322	12·91	4·02	8·89	12·77	—	12·79	12·80
February ...	1·0323	12·84	3·95	8·89	12·72	—	12·75	12·83
March ...	1·0322	12·74	3·88	8·86	12·72	—	12·73	12·72
April ...	1·0322	12·54	3·72	8·82	12·46	12·31	12·44	12·49
May ...	1·0323	12·54	3·70	8·84	12·49	12·41	12·42	12·50
June ...	1·0322	12·42	3·62	8·80	12·32	12·25	12·24	12·40
July ...	1·0316	12·47	3·74	8·73	12·39	12·33	12·34	12·44
August ...	1·0315	12·57	3·89	8·68	12·48	12·41	12·45	12·48
September	1·0316	12·71	4·00	8·71	12·67	12·56	12·67	12·68
October ...	1·0318	12·94	4·16	8·79	12·81	12·72	12·73	12·81
November	1·0318	13·03	4·23	8·80	12·79	12·76	12·78	12·85
December...	1·0318	12·80	4·02	8·78	12·61	12·63	12·76	12·73
Average	1·0320	12·71	3·91	8·80	12·60	(12·54)	12·59	12·65

During the first three months of the year the quality of the milk was fully up to the average, but in the later months a depression, especially in solids-not-fat, was noticed. This causes the yearly average to be the lowest yet observed, lower even than last year. A remarkable instance of low solids-not-fat has already been brought under the notice of the Society (ANALYST, xviii., 4.) In this case I have shown that the low solids-not-fat was due to the milk sugar being below the normal quantity; and I may take this opportunity of expressing my opinion that it is important in cases of abnormal milk to make as full an analysis as possible, as notable differences from the normal proportion of some particular constituent may be detected, and the sample thus deprived of its value to the unscrupulous chemist who defends adulteration cases on the principle expressed in the proverb "*se non è vero, è ben trovato.*" The differences between the five series of analyses is rather more marked this year than in previous ones. An explanation of these differences is to be found in a paper by Dr. Vieth (ANALYST, xvii., 86). As usual, the highest percentages of total solids and fat are to be found in November, while, as is frequently the case, the lowest occur in June.

Cream samples were taken for analysis before and during delivery. The average of the results is given in the following table :—

AVERAGE AMOUNT OF FAT IN CREAM.

	Before Delivery.		During Delivery.	
January	46·3	..	46·0	
February	47·1	..	47·5	
March	47·0	..	47·3	
April	48·3	..	48·9	
May	48·3	..	48·9	
June	45·9	..	46·5	
July	48·5	..	49·5	
August	46·0	..	47·3	
September	46·6	..	47·4	
October	47·1	..	47·1	
November	44·3	..	43·9	
December	44·2	..	44·1	
Average	46·7		47·0	

The average composition of Clotted Cream was as follows:—

Water	35·63 per cent.
Fat	56·27 „
Proteids and Milk Sugar ..	7·52
Ash	·58

Skim Milk produced by centrifugal cream separators contained, as a rule, from ·2 to ·4 per cent. of fat.

Butter had the following composition:—

French Butter, fresh:—

Fat	85·49 to 83·12.	Average 84·39
Water	14·93 „ 13·29.	„ 13·98
Solids-not-fat ..	2·22 „ 1·09.	„ 1·63
Including salt ..	·18 „ ·07.	„ ·12
Reichert-Wollny Fig.	30·8 „ 25·4	„ 29·1

French Butter, salt:—

Fat	85·35 to 81·93.	Average 83·44
Water	14·32 „ 11·29.	„ 12·86
Solids-not-fat ..	4·29 „ 2·73.	„ 3·70
Including salt ..	3·08 „ 1·26.	„ 2·07
Reichert-Wollny ..	32·8 „ 26·2	„ 29·1

English Butter, salt:—

Fat	86.19 to 80.14.	Average	82.98
Water	16.49 „ 11.58.	„	13.99
Solids-not-fat	4.98 „ 1.78.	„	3.03
Including salt	3.93 „ 1.16.	„	2.14
Reichert-Wollny	30.8 „ 24.9	„	28.1

A few odd samples of Danish and New Zealand butter were analysed; the Reichert-Wollny figure of the latter were high, being in three samples 32.1, 31.7, and 32.8. These figures are not remarkable, but they show that the large amount of volatile fatty acids in antipodeal butter, which has been remarked by several observers, seems to be normal. Nothing abnormal has been noticed this year; but the search for these samples has not been so thorough as in previous years. The samples giving the lowest Reichert-Wollny figures were prepared from the milk before referred to as being abnormally low in solids-not-fat. Two samples of this butter gave 24.9 c.c. of N/10 alkali consumed.

An opportunity occurred of studying the changes that occur on freezing milk. The samples under notice are remarkable, as the ice and the liquid portion show a greater difference than has hitherto been observed. Their composition is as follows:—

				Ice.	Liquid.
Water	96.23	85.62
Fat	1.23	4.73
Sugar	1.42	4.95
Proteids91	3.90
Ash21	.80
Specific gravity	1.0090	1.0345

The quantity of ice amounted to about 10 per cent. in this case; the proportions that the various constituents bear to each other is not markedly different in the ice and the portion which is unfrozen, showing that no great separation, if any at all, has taken place during freezing.

A number of comparisons have been made during the past year between the Adams and Werner-Schmid methods of fat-estimation; the result has been practically absolute agreement. As the Werner-Schmid method, as worked in the laboratory under my charge, differs in some respects from the usually published methods, I think it of interest to give a short description; it is exactly the same modification that I worked three years ago in Egypt, and consists in taking 5 c.c. of milk in a stoppered tube (I now use a Horsley's tube) and diluting with 5 c.c. of water; about 11 c.c. of strong hydrochloric acid are added, and the whole boiled over a naked flame till the fat forms a clear layer on the top, the tube being constantly shaken during the boiling. Immediately the boiling is completed the tube is cooled. About 25 c.c. of ether are then added and the whole well shaken and allowed to settle. The separation of the two layers is practically instantaneous, and no fluffy-looking layer appears at the junction of the ethereal and aqueous strata; as

much as possible of the ether is pipetted off, and a further quantity is added, and the treatment repeated three times over; the ether is evaporated and the fat weighed. This method, for the introduction of which into this country chemists owe a debt of gratitude to Mr. A. W. Stokes, has the advantage over the Adams method that the fat can be estimated within an hour or so of the receipt of the sample, in those rare cases where this is necessary; in this, however, it stands inferior to the Leffmann-Beam process. For convenience it is, in my opinion, markedly inferior to the Adams method, and involves more labour where many samples are done. This method has been claimed to be more theoretically perfect than the Adams process by Mr. Stokes (ANALYST, xvi., 71); as the Adams method is after all our official standard process, I may be pardoned for pointing out the fallacy of the reasoning of this chemist. He states that the most perfect medium for extraction is a gas, the next a liquid, and last a solid stands in order. The conditions that decide the suitability of a medium for the purpose of fat-extraction are—penetrability, insolubility of extraneous substances, and immiscibility; obviously, if the solvent and the medium are in two different states their immiscibility is better ensured than in the contrary case; two liquids possess, perhaps, slightly more penetrability than a liquid and a solid, but in the case of any reliable method of fat estimation from solids, this advantage is practically nil; the solubility of extraneous substances is decidedly greater in a case such as the Werner-Schmid method; the advantage rests on theoretical grounds with the Adams and similar methods; the extraction from a gas is obviously much more difficult than from either of the other states of matter, and it surprises me that such a statement should have been allowed to pass unchallenged. In showing the fallacy of Mr. Stokes' reasoning, I do not wish to unduly disparage the Werner-Schmid method, but my intention is to contest the assertion that the Adams method is inferior. The Werner-Schmid method gives very good results indeed, but it is scarcely so reliable as the Adams process.

The estimation of total solids has been much studied during the past year; a slight modification of Babcock's method has already been described (ANALYST, xvii., 225); this method has been used to a considerable extent. The results by this method have been a little higher than by the ordinary process; the actual proof that this is more correct than the usual process is not yet complete, but the following indications of this may be adduced: 1. The agreement between the fat found and that calculated by the "milk-scale" is relatively constant, a variation of 0.2 per cent. covering the whole of the experiments. 2. Complete analyses, in which water, fat, milk-sugar, proteids, and ash are estimated, add up to about 99.8 per cent. One would naturally expect an analysis comprising the constituents enumerated to add up a little below 100 per cent., on account of the presence of traces of lactic acid, urea and other bases, salts in the milk not included in the ash, &c., and from our present knowledge of the composition of milk, 0.2 per cent. is a very reasonable approximation of the quantity of these unestimated bodies.

A very extensive investigation, comprising 2,930 analyses, was made regarding the

rising of cream in the churns in which milk is taken out for delivery; it would be impossible to give here the results of all the analyses made, but the averages are to be found in the first table in this paper. The general conclusion was that the tendency of cream to rise was overcome by the constant slight agitation in transit, provided that at no time the rising of the cream was allowed to commence by a prolonged standing; if this had been allowed to take place the shaking was not sufficient, and the cream steadily rose; even in rounds which were out for six hours no rising of cream could be detected, provided that no long intervals of time were permitted in which the milk remained at rest.

A series of comparative experiments was carried out on the relations between the Reichert-Wollny and Leffmann-Beam methods of butter analysis. The relations were:—Reichert-Wollny : Leffmann-Beam = 2.20 : 1 (ANALYST, xvi., 153). In the Leffmann-Beam method only 2.5 grammes were taken and 50 c.c. distilled out of 75 c.c. The ratio between these proportions, alcohol being used in both, has already been found to be 2.23 : 1 (ANALYST, xviii., 17). This shows that the glycerol saponification gives slightly higher results than the alcohol saponification, and this has been confirmed by direct experiment. The use of glycerol has several distinct advantages, among which may be enumerated sharper end reaction, clear distillate, absence of possibility of loss of ethers during saponification, and saving of time.

DISCUSSION.

Mr. Allen said that when it was considered how small a part of his work Mr. Richmond had shown in the figures which he had placed on the board, it would be easily seen how extremely indebted the members of the Society must be to him for laying before them the annual average results of all the samples of milk analysed by him. Mr. Richmond's experience of the Leffmann-Beam process of estimating volatile fatty acids, as distinguished from the Reichert process, was of much value. He would remind the members that the Leffmann-Beam method consisted in saponifying with a solution of alkali in glycerin, by which means the tendency to the formation of butyric ether was avoided. He (Mr. Allen) had used the Leffmann-Beam process, and he found it gave results somewhat *higher* than the Reichert. It was, no doubt, a question of manipulation, for the American Association of Official Chemists reported exactly the contrary, namely, that the Leffmann-Beam results were lower rather than higher. He was glad to see that the figures obtained by the Reichert-Wollny process fell within reasonable limits. He was also interested in noting that the milk yielded by antipodean cows contained a higher proportion of fatty acids than was the case on this side of the globe.

With regard to Dr. Adams' coil process of determining fat in milk and the Werner-Schmid process, he had of late been using the Werner-Schmid process, because it was less troublesome, and it had been proved that the two processes gave substantially identical results. If two processes gave the same results, he did not see why the simpler one should not be used. The coil method was the official method of the Society; but it was

possible that evidence might be adduced in the future which might induce analysts to abandon it, or replace it by another process; and he could not endorse the view which Mr. Richmond apparently held that an analyst who used any other than the official process was a sort of traitor to the Society.

The total proportion of water found in butter was of interest as showing that the opinions held by public analysts, who had considered the matter, were well borne out; the only instance where it came over 16%, being in an experimental sample. He had very strongly laid down in the witness-box and elsewhere that 15% was the maximum quantity of water to be allowed in butter, which could be raised to 16% as an outside amount. He thought a rigid line should be drawn at 16 %, and he believed that if that were done there would be very little difficulty in reducing the quantity of water in commercial butters to that amount. Some people could, no doubt, be found who would excuse even 35 or 40 per cent.; and an inspector from the Cork butter market recently stated on oath that the proportion of water in the butter depended on the atmospheric conditions at the time the cows were milked. Similarly, Dr. Bell had recently stated that he did not see his way to regard butter as adulterated if it did not contain more water than had been known to be left in it, when not manufactured for sale. This seemed to him to be practically making an incompetent dairy-maid the referee under the Food Act.

Mr. Hehner asked Mr. Richmond for particulars with regard to solids-not-fat. The average had been given, but he (Mr. Hehner) would like to have the extremes, and to know how many abnormally low results had been recorded, if any. He thoroughly agreed with Mr. Allen that 15 per cent. was a reasonable and liberal figure to allow for water in butter. It was, he believed, a fact, that in Denmark many samples had been recently met with giving upwards of 15 per cent. As Public Analysts, they were likely to meet with the contention that, on the authority of Professor Stein, butter contained very frequently more than 15 per cent. of water. It must be borne in mind that such samples should not be compared with the samples submitted to Public Analysts, especially in England. The butter lost a notable amount of water in travelling. The limit of 15 per cent. was calculated on trade samples of butter, and not on butters that came directly out of any dairy. All sellers of butter knew that almost every keg of butter lost a certain percentage in weight from exudation of water; and most butter merchants did not pay on the original weight as shipped from abroad, but on the actual weight received by them. It was very important to bear in mind that samples coming straight from the dairy would, in the majority of cases, contain more water than butter as sold to the public or submitted to the analyst.

Mr. Woosnam said that his own experience agreed with that of Mr. Richmond with regard to the distribution of the solids in the case of frozen milk. He had found that it was very difficult after thawing to get a fair sample of the milk as originally produced; and he did not think that by passing through a sieve the chance of abnormal samples was appreciably lessened. The solids—which collect on the bottom of the churn, in a layer

when the milk freezes—are with great difficulty re-dissolved, and would, to a large extent, again settle out after passing through the sieve, leaving the supernatant fluid nearly as poor as before. He (Mr. Woosnam) used the Werner-Schmid process a good deal, and the Adams process for the purpose of checking. He did not pipette off his fat solution, but used a small blow-tube with a mouthpiece attached, as in the ordinary wash-bottle.

Mr. Richmond, referring to Mr. Woosnam's remarks, said that the proper course to follow was to strain the ice off and throw it away, it being much safer than thawing. Thawing was certainly the right course to follow if there was only a choice between doing that and sending out first the liquid milk, and then the liquid produced by thawing the ice. With regard to the question asked him by Mr. Hehner, he could not give the exact figures, but he could give an approximation. Out of 13,196 samples received from the country there were 34 yielding less than 8.5 solids-not-fat. 29 came from the farm to which he had already drawn attention. He could not say how many milks were above 9 per cent., but there was only one above 9.5. The abnormal samples were thus very few. As to the question of water in butter, the sample which gave 16.49 per cent. was churned under special conditions, and it was submitted at once to examination. He thought that his results showed that 15 per cent. was certainly a sufficiently high amount to allow. He thought he was perfectly right in maintaining that the Adams process was a correct one. It was the standard process of the Society of Public Analysts, and if they used any other method it was always compared with that. The only claim the Werner-Schmid process had to accuracy was that it gave results which compared well with those of the Adams process. If he wanted to get an idea as to the fat in a sample in a short space of time he used the Leffmann-Beam process, which gave results sufficiently near for such a purpose.

Mr. John Hughes asked Mr. Richmond whether the results of his 23,000 samples were obtained by the Adams or the Werner-Schmid process?

Mr. Richmond replied that the bulk were obtained neither by the Adams nor the Werner-Schmid process, but by the fat-calculation process. The method had been fully explained by Dr. Vieth in former papers; any particular sample was not experimentally exact, but when the average of some thousands was taken, it was not far out.

Mr. Cassal, referring to the sample containing 8 per cent. of solids-not-fat, asked Mr. Richmond if he considered that the term "genuine milk" could be applied to that sample, and whether he would regard it as being of the "nature, substance, and quality" of milk?

Mr. Richmond stated that in his own particular case he had certain standards to work up to. That particular sample did not come up to that standard, therefore he reported it as such, and the necessity for the fine distinction drawn up by Mr. Cassal did not arise.

Mr. Cassal said his object was to elicit from Mr. Richmond whether he would condemn a sample containing 8 per cent. of solids-not-fat; and while he did not doubt that Mr. Richmond would do so, he (Mr. Cassal) thought it advisable that the fact should be definitely stated. To have a standard to work up to was equivalent to having a rigid definition. He again pointed out that any sample containing so low an amount as 8 per cent. of solids-not-fat was not "milk."

Mr. Allen considered that the analyses of 23,000 samples in the course of a year was a remarkable experience, and that experience was especially valuable when it was added to the 120,000 samples analysed by Dr. Vieth. He had had occasion lately, in Court, in a skimmed milk case, to refer to the valuable statement compiled by Dr. Vieth some time ago; and when the magistrates were told that in these milks the fat ranged from 3.8 to 3.6 per cent., and that the milk in question contained 2.2 per cent., they were much impressed.

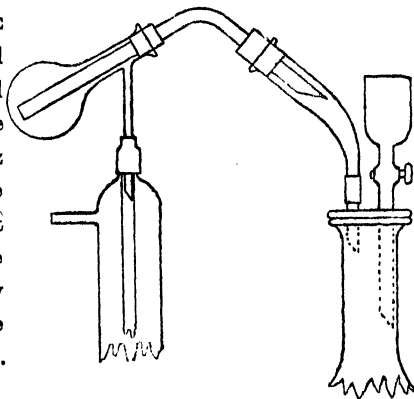
In the absence of the author, Dr. Dyer read the following papers:—

THE "STOCK" PROCESS FOR THE DETERMINATION OF NITROGEN.

A MODIFIED DISTILLATION APPARATUS.

By W. F. KEATING STOCK, F.C.S., F.I.C.

THE apparatus hitherto used was described in THE ANALYST for June, 1892 (p. 110.) I have dispensed with one of the two Bunsens there shown, and have given the apparatus the form seen in the accompanying sketch. It consists of a Wurtz flask of not more than 150 c.c. capacity, the tube from which runs into the condenser, and it is kept at boiling point by the ammoniacal steam from the distilling flask. I have found that it entirely prevents the conveyance of sodium hydrate into the distillate, and that it does not retain ammonia. It is a very great gain in simplicity.



THE STOCK METHOD FOR THE RAPID DETERMINATION OF NITROGEN IN ORGANIC BODIES.

A REPLY TO MR. W. P. SKERTCHLY BY W. F. KEATING STOCK.

At the May Meeting of this Society I had the honour of introducing to your notice a "New and Rapid Method for the Determination of Nitrogen in Organic Bodies," in a paper which will be found in THE ANALYST (June, 1892, pp. 109 to 113). During a discussion which followed the reading of my paper, attention was directed to a possible loss of nitrogen by progressive oxidation through ammonia on to nitric acid or even free

nitrogen. At the special meeting held in June, I submitted a reply to this objection, and gave the results of numerous experiments which showed that the feared loss of nitrogen did not occur. This supplementary paper was published in *THE ANALYST* (Aug., 1892, pp. 152 and 153).

At the meeting held in October, Mr. W. P. Skertchly read a paper on my process in which he gave a long series of test-analyses which I feel bound to say do him much credit. He proved by these analyses that up to a certain point he could obtain most excellent results over a variety of material, including, like my own test analyses, pure ammonium salts and nitrogenous organic matter. He, however, arrived at a point where, in his hands, the process broke down and failed to yield the whole of the nitrogen he knew to be present in certain samples. *THE ANALYST* for November contains Mr. Skertchly's paper. Arguing from the results of his experiments, Mr. Skertchly and another member (Mr. Perry Coste) sought to explain by a number of propositions that the failure was directly due to a *loss* of nitrogen in one form or other, and held that until some preventative of the said loss could be discovered, analysts could not rely upon its performance.

I could not see my way to accepting the explanation of Messrs. Skertchly and Perry Coste as being a satisfactory settlement of the difficulty, and I felt diffident about recognising the existence of the several varieties of nitrogen which they suggest might exist in the substances which had proved to be so puzzling. I therefore wrote to Mr. Skertchly and asked him to favour me with duplicates of his samples. This he very courteously and promptly did, and I am now in a position to place my own analyses before you. The samples I received from Mr. Skertchly consisted respectively of Crushed Hoofs and Horns, Fish Guano No. 2, Dried Blood and "Manure." A preliminary inspection revealed the fact that they were by no means so finely divided as I thought desirable for this class of material. The Dried Blood was especially faulty in this respect. I, however, made a trial analysis of this sample in the condition in which it came into my hands. I got 12.90 per cent. of nitrogen as against Mr. Skertchly's 10.82 per cent. I now reduced all the samples by milling, until they passed a 36 sieve, and then determined the nitrogen in each exactly as described in my original communication. The results obtained are given below, side by side with Mr. Skertchly's own figures for his results by my process, and also with those got by himself and Dr. Dyer by the modified Kjeldahl process:—

	THE STOCK PROCESS.			THE MODIFIED KJELDAHL PROCESS.		
	<i>Stock.</i>	<i>Skertchly.</i>		<i>Skertchly.</i>	<i>Dyer.</i>	
<i>Crushed Hoofs and Horns.</i>						
Nitrogen, per cent. ...	14.95 ...	13.37 ...		15.34 ...	15.38	
" " ...	15.09 ...	13.30 ...		— ...	—	

Fish Guano, No. 2.

Nitrogen, per cent.	...	5.71	...	4.97	...	5.50	...	5.60
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" "	...	5.71	...	—	...	—	...	—
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Dried Blood.

Nitrogen, per cent.	...	13.18	...	10.81	...	12.93	...	13.08
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" "	...	13.12	...	10.82	...	—	...	—
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Manure.

Nitrogen, per cent.	...	6.24	...	5.56	...	6.16	...	6.09
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" "	...	6.27	...	—	...	—	...	—
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Here, then, is the true solution of the problem which called forth Mr. Skertchly's very ingenious but groundless "explanations." The simple fact is that, so far from his having *lost* nitrogen, he had, owing in the first place to want of division of his samples, left it unconverted. I say, in the first place, because it is evident on perusal of Mr. Skertchly's paper, that he had taken a good deal of liberty with the mode of procedure laid down by myself. Take, for example, the composition of his oxidant. I prescribe manganese dioxide; Mr. Skertchly uses a substance containing, by his own showing, only 40.86 per cent. of MnO_2 . What the remainder was I do not know; but I do know that I can get native manganese dioxide, which shows by analysis from 80 to 97 per cent. of MnO_2 .

If I am asked to explain why, in respect of these four samples, my process failed where the modified Kjeldahl succeeded, I answer that Mr. Skertchly did not use *my* process but a bad copy of it, and that he was wrong in the three essential particulars of purity of reagents, condition of sample and relation of quantities. Time is a very important item in any form of Kjeldahl's process. My process is what it professes to be, a *rapid* process, and it also proves to be possessed of a much more important quality, that of accuracy.

DISCUSSION.

Mr. Allen said that a paper of this description was necessarily of a controversial nature, but it was obviously important that any process they employed could be depended on to give correct results. In working the Kjeldahl process he had found it desirable to make two or three determinations and take the highest result. It appeared that some experimenters had abandoned the use of permanganate, and used Gunning's modification, namely, the addition of sulphate of potassium to the acid, with the view of increasing the temperature. Then sometimes, mercury was used and again there were other modifications. Analytical chemists in America had been making a number of experiments with the Kjeldahl process, with and without permanganate, and with and without sulphate of potassium. The results showed that there was no difference in the amount of ammonia obtained. Mr. Stock's method was eminently interesting. It seemed to give results which were fairly in accordance with the ordinary Kjeldahl process. One man did not work under the

same conditions as another, and what would be a most convenient process for one might be very inconvenient for the other.

Mr. W. Pearson Skertchly said that he had followed Mr. Stock's method as nearly as he could. It was only after taking the small quantities of the substance used by Mr. Stock that he tried larger amounts, and the results so obtained were more satisfactory than in the former case. Of course, he increased the quantities of sulphuric acid and of manganese dioxide proportionally. All his substances were actually dissolved in the sulphuric acid before the dioxide was added, and he therefore did not think that Mr. Stock had given a good reason for the solution of his (Mr. Skertchly's) difficulty. Mr. Stock in his first paper said that the *manganese dioxide* should be passed through a sieve, having 36 meshes to the linear inch, and in the paper just read he said that the *substance* ought to pass through such a sieve. He did not write his paper with the intention of fault-finding, but merely to show that as he (Mr. Skertchly) had failed with the method, others should not rely on it until it had been proved to give satisfactory results in every case. Personally, he would only be too glad if a quicker process than the Kjeldahl were devised.

Mr. Hehner did not think that Mr. Stock had made out a very strong case for himself. Mr. Stock concluded, from the appearance of the samples given him by Mr. Skertchly, that they were not so finely divided as he deemed desirable, but when he subjected the very first sample, without subdividing it further, to his process, he obtained a result which tallied with that obtained by the Kjeldahl process, but which did not agree with the results obtained by Mr. Skertchly. In spite of this fact, Mr. Stock came to the conclusion that he must more thoroughly subdivide all the other samples. Now, as Mr. Skertchly dissolved the samples first in the sulphuric acid, that disposes entirely of Mr. Stock's explanation of the differences. It was certainly true that Mr. Skertchly had used a bad sample of manganese dioxide, but it appeared to him (Mr. Hehner) that it made no difference whether the manganese was inferior or not, provided it contained sufficient dioxide to oxidise the substance. Mr. Skertchly had added manganese until a green colour was produced, and Mr. Stock considered this to be the criterion of complete oxidation. It was a remarkable fact, that at first Mr. Skertchly obtained good results, and afterwards bad ones; but Mr. Stock had himself, in his earlier experiments, experienced a similar difficulty in obtaining the total nitrogen from potassium ferrocyanide; and though he stated that he had eventually succeeded with that salt, he did not furnish the explanation of his previous failure. When his process was repeated in duplicate it gave agreeing results, but these differed entirely from those obtained by the Kjeldahl process. If precisely the same figure was obtained, he could not imagine how the differences referred to could be ascribed to insufficient subdivision. Mr. Hehner was far from satisfied that even yet Mr. Stock had laid down the exact conditions under which his process would work in every case.

Mr. F. H. Perry Coste concurred with Mr. Hehner in thinking that Mr. Stock had not made out a strong case for himself. As to Mr. Stock's advice that they (Mr. Coste and Mr. Skertchly) should suspend judgment on the matter, that was precisely what he had said. He had spoken very sympathetically of Mr. Stock's process; but it seemed clear that if anyone but Mr. Stock used the process, it was not applicable to ordinary working at present. If other chemists could not obtain the same results, the process required further investigation. He (Mr. Coste) had spoken after deliberate experiment on the behaviour of permanganate. Some time ago, in order to clear up a point in the Kjeldahl process, he had made experiments which proved conclusively that boiling with permanganate of potash did cause a loss of ammonia. He observed that in one determination Mr. Stock had obtained 14.95, as against 15.38 by the Kjeldahl process, a difference of over .4. It would be interesting to know whether that analysis was made by his own method, or by the *bad copy*, as he called it, which Mr. Skertchly had used. One disadvantage of the Stock process was that the samples had to be finely ground. With regard to Mr. Allen's remarks, experience with the Kjeldahl process showed it to be one of the most accurate in the world; and when one was used to it, it acted perfectly. As to the sulphate of potash in the Gunning process, it undoubtedly was very useful; he had always used it with mercury. The process could be worked without mercury, but it took far longer. With mercury and sulphate of potash three-quarters of an hour was an outside limit even with gelatin.

Mr. E. J. Bevan was very much interested in Mr. Stock's process, partly for the reason that some years ago, in conjunction with Mr. Cross, he had brought out a process for estimating carbon in organic substances by treatment with a mixture of strong sulphuric acid and chromic acid as an oxidizing agent. It had occurred to them that they might be able to estimate the nitrogen in the same way, and they had made some experiments with nitrogenous substances, the results of which were as follows:—

Substance.	Weight taken.	Conditions.	% N. found.	True Percentage.	
				Soda lime.	Kjeldahl.
Gelatin	0.286	CrO ₃ added at once	12.92	17.60	17.62
Do.	0.302	Do. do.	12.80	"	"
Do.	0.586	Do. previously dissolved, in H ₂ SO ₄	12.80	"	"
Do.	0.271	Do. do.	12.95	"	"
Do.	0.228	H ₂ SO ₄ only	none	"	"
Dried blood	0.273	CrO ₃ added at once	9.22	11.64	—

It was somewhat remarkable, that, whilst the percentages obtained were nothing like the actual proportions present, the concordance was very good, although the conditions and the amounts taken varied considerably. In one experiment he got no ammonia at all. In that case he used only sulphuric acid—he did not use any chromic acid—and the result seemed to him to be remarkable, in comparison with the results obtained by the

members of the American Society. In any substance containing nitrogen, when that substance contained a large amount of carbon, there was no doubt that a certain amount of ammonia was formed by the action of sulphuric acid. 0.273 grammes of dried blood gave 9.22 per cent. of nitrogen, the actual amount found by the soda lime process being higher. After seeing an account of Mr. Stock's process in THE ANALYST, he (Mr. Bevan) tried the action of a mixture of sulphuric acid and chromic acid on pure sulphate of ammonia. No ammonia was lost by the action, so the difference had still to be accounted for. He had not examined the product from this reaction, so that he could not say into what form the nitrogen had gone, but it did not go into the form of ammonia. He could not help thinking that it was a pity Mr. Skertchly had not followed the exact details given by Mr. Stock. It was hardly fair to criticise and condemn a process when one worked with four times as much substance as the author suggested. It might possibly be that it was important to take a small quantity. Although he agreed with Mr. Hehner that if complete oxidation was obtained, the actual percentage of manganese dioxide in the sample used should not, theoretically, make any difference, still there might be some practical difference; and he thought Mr. Skertchly owed it to himself and to Mr. Stock that he should make the experiments under exactly the same conditions, using the same quantities, and a similar manganese to that used by Mr. Stock.

Mr. John Hughes wished to mention his own experience of Mr. Stock's process. So far as he had gone he had not been able to get the full amount of nitrogen. He quite agreed with what Mr. Hehner had said in reference to the coarseness of the substance. It was a matter which did not require any further argument, because the figures given in the paper obtained equally good results with the coarse, as with a fine substance. He hoped that Mr. Stock would give the members of the Society a little fuller information, and then, perhaps, its excellence would be generally testified to, like the excellence of the ordinary Kjeldahl process. His experience was similar to Mr. Allen's—he could not get the full results which Dr. Dyer and Mr. Coste found no difficulty in obtaining. It might be due to want of skill in manipulation, and he thought the same remark probably applied to Mr. Stock's process. It might eventually prove the better of the two processes.

Mr. Allen agreed with Mr. Bevan that strict adherence to the prescribed method of an author was proper and necessary when making experiments by his process; though he could not see why a process which gave good results on $\frac{1}{4}$ or $\frac{1}{2}$ a gramme should not give the same results on 2 grammes. Mr. Bevan's results were highly suggestive. In one of his experiments he had no doubt formed certain amido-acids. Did not his results tend to show that gelatin contained distinct groups, one of which yielded ammonia direct, another by the influence of oxidising agents, while a third was not converted into ammonia; if so, this would seem to throw doubt on the Gunning process. The subject was one which would well repay further attention and experiment.

NOTE ON THE THEORY AND PRACTICE OF THE REICHERT PROCESS.

By H. DROOP RICHMOND.

(Read at the Meeting, November 2nd, 1892.)

In a recent number of the *Chemical News* (October 21st), Mr. J. A. Wilson calls into question the accuracy of the Reichert process; in the former paper (*Journ. Soc. Chem. Ind.*, ix., No. 1), he had shown that the rate of distillation of acetic acid was influenced by the salts in the solution distilled, both the nature and the quantity of the salt tending to vary the result; and he argues from the analogy between acetic acid and the acids of butter that the same holds good.

As the Reichert process is now one of our standard methods, it is very important to show that however scientifically correct Mr. Wilson's view may be, in practice the error is quite negligible.

In the *Stazioni Sperimentali Agrarie Italiani*, xxiii., No. 1, I have shown that butyric acid distills according to a parabolic formula; I have pointed out that the distillation is influenced by the amount of acid in solution and by the condensation of the vapour; the rate of distillation is a direct function of the strength of the solution, and the condensation is a direct function of the rate of distillation, or to put it more exactly, of the amount of acid vapour in the total vapour. Were Duclaux's logarithmic formula correct, the condensation would be an inverse function of the amount of acid in the vapour, which is absurd. I have calculated the formula $y = 2.22x - 0.0151x^2 + 0.000031x^3$ (x = quantity distilled from 100 c.c., y = per cent. of butyric acid in the distillate) for the distillation of butyric acid in the Wollny apparatus. No logarithmic formula can be found to express the results with any degree of accuracy.

From this formula I calculate that when 110 c.c. are distilled from 140 c.c., that 96.3 per cent. of the total acid should pass into the distillate; and that when 50 c.c. are distilled from 75 c.c., that 89.1 per cent. should be found in the distillate.

I find, when these proportions are distilled with the addition of the quantities of alkali and acid used in the Reichert-Wollny or the Reichert processes, that I get 93.9 or 89.6 per cent. (uncorrected for impurities in the alkali and acid); and further when 4.4 or 2.2 grams. of well-washed fatty acids are added, that the figures are 97.2 and 89.7 per cent. respectively.

These figures show that for butyric acid the amount of salts in solution in the Reichert process have no practical influence on the results. My results also seemingly controvert Wollny's statement that the fatty acids hold back the volatile acids; they do not really do so. As Wollny's statement is loosely worded, he actually only shows that

the fatty acids, when not completely decomposed (so-called "melted") or when solid, hold back the fatty acids.

As the higher fatty acids distil even more readily than butyric acid, the conclusions drawn from the latter apply with more force to them.

I have experimented with Leffmann and Beam's modification in which glycerine is used, and thereby the boiling point is raised; the excess obtained (for 2.5 grams. 0.07 c.c.) by this modification is much less than the experimental error.

Mansfeld (*Milch-Zeitung.*, 1888, xv., 281), shows that by the use of potash instead of soda no difference in the results is obtained, and this has been confirmed by many other observers.

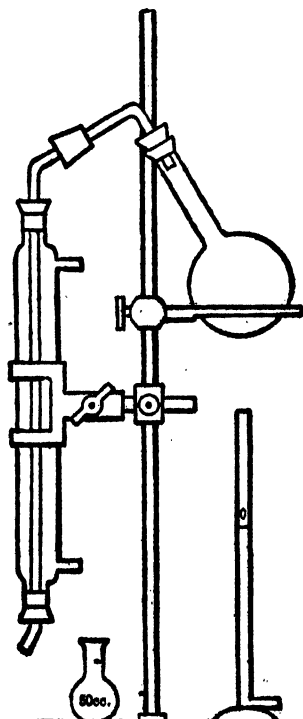
Mr. Wilson's objections are from a practical point of view groundless, and are based on the assumption that the volatile fatty acids of butter, distilled in a particular way, are influenced by certain external influences to the same degree as acetic acid is when distilled in another way. A study of the laws of the distillation of these acids will show that scientifically he is correct when he alleges errors, but that these errors are so small as to be negligible.

APPARATUS FOR THE ESTIMATION OF FREE AND ALBUMINOID AMMONIA IN WATER ANALYSIS.

By AUGUSTUS H. GILL, PH.D.

THE article by Mr. Embrey* upon this same subject, has induced me to describe the apparatus for these determinations in use in the laboratory for water analysis of the Massachusetts Institute of Technology, used by the State Board of Health.

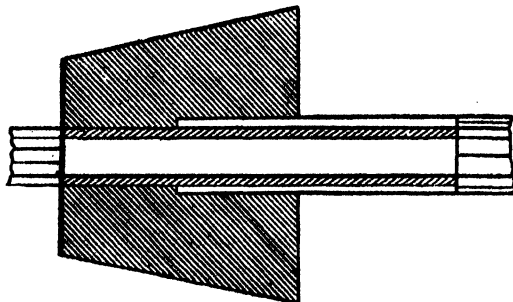
The arrangement will, I think, be evident from the accompanying sketch. The apparatus consists of a nearly spherical flask with square shoulders, of 850—900 c.c. capacity, connected by a bent glass tube and "cork joint" with a vertical block tin condenser. The "cork joint" is here shown in section, and consists of a sound cork fitted at one end with the condenser, and at the other, with the glass tube, indicated by the cross hatching, which enters the former for about an inch-and-a-half, thus avoiding all contact of steam with the cork, and making a



* The Analyst, xvii., 41.

tight and durable connection; the flask is closed with a superfine cork carrying the bent glass tube.

The condenser is the usual glass jacket, with the inside tube of block tin, three ounces to the foot, one-quarter inch internal diameter, twenty-four inches long and bent at the top for the "cork joint," and at the bottom for delivery into the fifty c.c. graduated flasks.† The lamps used are ordinary Bunsen burners lengthened by inserting a piece of pipe between the base and the burner proper. The whole apparatus is supported by the ring and clamp upon a brass rod fixed into the distilling table. This table is eighteen



CORK JOINT.
Full Size.

feet long, two feet wide, and three feet high, with two thirteen-inch sinks near each end; upon it are arranged the two rows of brass rods five inches apart, with eighteen inches between each rod, thus affording space for fourteen sets of apparatus, and making the laboratory equipment capable of handling sixty to seventy samples per day. All the piping is underneath the table, the rubber tubing passing through it, one set of tubing being painted black to distinguish the rows; each condenser and lamp is provided with its own stopcock, or the whole can be turned off together.

For designating the number of the water analyzed, slips of ground glass are placed between the vertical rods.

The apparatus is distilled free from ammonia before each determination, and, as we have waters that show neither free nor albuminoid ammonia, it indicates that the working is satisfactory.

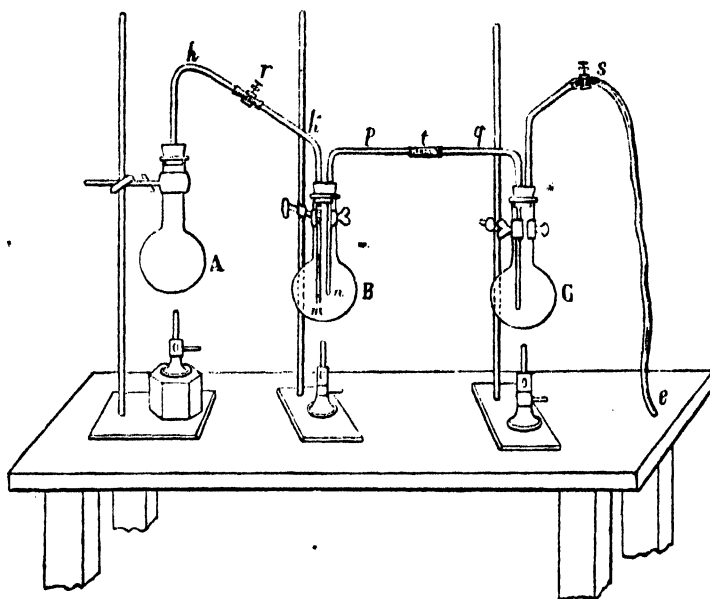
The condensation, even in the hottest weather, is absolutely perfect. Spiral condensers of glass and tin have been used, but were too fragile and cumbersome to admit of their continuance.

With this arrangement one person performs fourteen distillations simultaneously in about an hour, the work all being done from one side. The present form has been in use eighteen months, during which time about 5,000 determinations have been made, which is sufficient proof of its practicability.

† Flasks are here used, as the Nessler tubes are inconvenient to manage; those in use hold 50 c.c., are 8 inches long and $\frac{1}{8}$ inch internal diameter, admitting of the reading of 0.000001 gm. of ammonia with absolute certainty.

Observations on the methods in use for the determination of the quantity of gas dissolved in potable waters. G. Musajo. (*Staz. Sper. Ag. Ital.* xxiii. 113.)

—In the determination of the amount of gas dissolved in water there are several sources of error, which arise from the entrance of water into the vessel in which the gas is measured; these are the solubility of the gas in the small quantity of water in the tube, and the oxidation on the mercury in the presence of water. Another source of error in the determination of oxygen lies in the absorption by pyrogallate of potash; either the absorption is not complete, or carbonic anhydride is liberated and measured with the unabsorbed gas. To avoid these errors the author proposes a new form of apparatus; this consists of three flasks arranged as shown in fig. 1. The flask A contains the water



under examination, and is closed by an indiarubber cork, through which passes the tube H; the extremity of this tube is drawn out, and is attached to the rest of the apparatus by an indiarubber tube, on which is a screw pinchcock. The flask can thus be filled and placed in position without risk of the water escaping.

The flask B acts as gasholder; it is closed by an indiarubber cork through which pass two tubes, as shown in figure. The obtuse-angled tube is divided in two parts, which are joined by an indiarubber tube just after passing through the cork. When joined the tube passes almost to the bottom of the flask; but if the upper portion is carefully withdrawn, the lower portion drops off, so that any gas in the flask may be completely expelled by the upper portion. The third flask is simply used as a reservoir. The apparatus is used as follows:—The flask B and C are nearly filled with distilled water,

and the corks are simply placed in them so as to allow steam to escape; the water in them is boiled for from 35 to 40 minutes; then the cork of C is inserted, and air is blown in till B is completely filled. The cork of B is next inserted, and this flask is then quite full. The two pinchcocks R and S are closed.

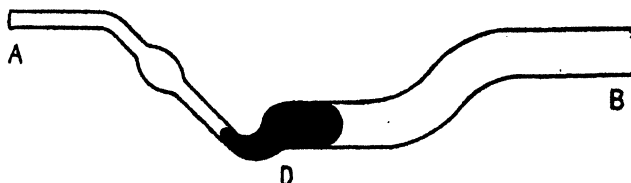
The flask A is filled with the water under examination; the pinchcocks are slightly opened, and air is blown in. When a small stream of hot water issues from the tube the drawn-out end of the tube H is inserted therein, and the apparatus is ready. The gas is lighted under the flask A, and under the flasks B and C when necessary to keep the water in them hot. The gas passes into B, and the displaced water flows into C. After forty minutes to an hour's boiling the pinchcock R is closed and the tube H withdrawn. The tube K is gently raised till the part D M drops off. The tube K can be emptied of water by allowing a flame to play on it for a second or two. A tube of small bore is inserted into the indiarubber tube, and the gas transferred through it into a measuring tube standing over mercury by blowing through the tube E till the water reaches the pinchcock R. No correction need be made for the small quantity of air in the small bored tube.

The analysis is performed by first pushing into the tube a piece of caustic potash, and noting the absorption. This is called carbonic anhydride. The author prefers pyrogallate of potash to cuprous chloride, sodium hydrosulphite, or ferrous sulphate and potash, as an absorbent for oxygen. He pushes up a ball of papier-mâché soaked in pyrogallate of potash, and repeats the treatment until no more is absorbed. He then uses a piece of caustic potash to absorb any carbonic anhydride formed in the reaction between oxygen and the pyrogallate. The residual gas is taken as nitrogen. All gases are reduced to the standard pressure and temperature. The remainder of this paper is devoted to the discussion of a few results obtained with saturated distilled water, and the water of his laboratory. In the latter the results for oxygen and nitrogen were those required by theory for a fully-saturated water, but the carbonic anhydride was notably deficient of the amount required to form bi-carbonates.

H. D. R.

Estimation of Oxygen in Lead. G. Lunge & E. Schmid. (*Zeit. f. Anorg. Chem.* 1892, II., 451 to 460.)—The shortness of re-melted lead has been attributed to the presence of dissolved oxide, and it has been stated that the resistance of lead to sulphuric acid depends on its content of oxygen. The authors therefore deem the determination of oxygen in lead a matter of some little importance. Their method is to pass hydrogen through the molten metal and weigh the water produced. The hydrogen from a Kipp's apparatus is passed through a wash-bottle containing caustic soda, one containing a solution of lead oxide in caustic potash (to absorb H_2S), two

containing silver nitrate solution, and two containing strong sulphuric acid; the gas then passes through a tube containing platinised asbestos, and heated in a furnace; this is followed by a wash-bottle of sulphuric acid and two U tubes containing phosphorus pentoxide. The hydrogen thus purified from the last traces of oxygen, is then passed through the molten lead contained in a hard glass tube:—



The hydrogen passes in at the end A, the metal is kept molten at D by a fan-shaped bunsen burner, and the water is caught by a phosphorus pentoxide U tube attached by means of a cork at B. A guard wash bottle of sulphuric acid completes the apparatus. The hydrogen is allowed to pass for two hours to free the apparatus from oxygen, and for another two hours after the platinised asbestos has been heated to redness. The authors made several experiments to prove the purity of the hydrogen at the end of the four hours, and are satisfied that this time is sufficient to ensure the absence of oxygen. The quantity of lead used should be under 30 grammes, and it, of course, must not be melted until the apparatus is absolutely free from oxygen. Half-an-hour is said to be sufficient to deoxidise the lead. The final U tube is then weighed with all the usual precautions.

The following figures may be quoted from the authors' tabulated results:—

						Oxygen Per Cent.
Firsts (Jungferblei)	0.00237
Do. alloyed with 0.02 per cent. of antimony					...	0.00363
Refined lead, alloyed with 0.02 per cent. of antimony						0.00250
Do.	do.	0.1	„	of copper	...	0.00343
Do.	do.	0.2	„	„	...	0.00566
Do.	do.	1.0	„	„	...	0.03661

Attention is called to the influence of copper in increasing the dissolved oxygen.

A. G. B.

Sulphurous Acid in Wine. M. Ripper. (*J. Prakt. Chem.* [2], xlv., 428—473.)—The author has made a searching investigation into the condition in which sulphurous acid exists in wines, and into the methods for its estimation; his experiments are fully detailed in the paper, which is of considerable interest.

Haas' method (*Berichte*, 1882, xv., 154), for the estimation in question consists in distilling the wine in a suitable apparatus, through which a current of carbonic dioxide is passed, and collecting the distillate in a bulbed U tube containing iodine solution; the sulphurous acid is thus oxidised to sulphuric acid which is weighed as barium sulphate. Desiring to use this as a standard process, the author sought for possible errors in it and alighted upon oxygen and hydrogen sulphide in the carbon dioxide as likely to influence the results; he states that the former gas is always present in carbon dioxide, made from marble and hydrochloric acid, and that the latter may in many cases be detected. These sources of error would tend to neutralise each other had they any real existence; that they have can hardly be allowed when the numbers obtained with carbon dioxide washed by water only, and by potassium permanganate and chromous chloride are compared. A more real source of error was found to exist in the impurity of the barium sulphate, upon which point the author has already written. (*THE ANALYST*, 1892, 233).

As an analytical process Haas' method is too lengthy and the author modifies it by collecting the distillate in caustic potash, acidifying this solution and titrating it with N/50 iodine solution. The details are as follows:—50 c.c. of the wine are pipetted into a distillation apparatus through which a current of hydrogen is passing. 5 c.c. of sulphuric acid (1:3) are added and the flask is heated in a glycerine bath at 80°—85° for three-quarters of an hour, the current of hydrogen being maintained the while. The sulphurous acid is absorbed by 20 c.c. of N-potash in the receiver, and after the time stated is liberated by the addition of 10 c.c. of sulphuric acid (1:3) and titrated with N/50 iodine solution, starch being used as an indicator. A number of experiments are quoted to show that no other easily oxidisable, volatile constituent of the wine passes over with the sulphurous acid. Experiments were then made to ascertain whether the direct titration of the sulphurous acid in a white wine be possible. With regard to red wines it may be said that they seldom contain sulphurous acid, inasmuch as their colour would be damaged by sulphuring; should an estimation be necessary it must be made by the distillation process. The fact that the iodine consumed by an acidified white wine is far below that necessary to oxidise the sulphurous acid present, as estimated by the distillation method soon brought the author to the conclusion that the sulphurous acid in wine is not all present as free acid or as alkaline sulphite. After many experiments it was found that aldehyde sulphite is the other form in which the sulphurous acid exists in the wine; this is not immediately oxidisable by iodine in presence of sulphuric acid, but as might be expected, is split up by the acid during distillation: it is, however, noteworthy, that the aldehyde and sulphurous acid in the distillate will again combine after a short time if no substance be present to remove one or the other. Very brief contact with caustic potash will decompose the aldehyde sulphite, and this opens the door for the determination of the *total* sulphurous acid in wine by direct titration. To this end 50 c.c. of the wine are pipetted into a 200 c.c. flask containing 25 c.c. of normal potash, the nose of the pipette being inserted into the liquid; after some 10—15 minutes 10 c.c. of sulphuric acid

(1 : 3) are added, and titration with N/50 iodine solution effected, starch being added as indicator. The *free* sulphurous acid is then determined by pipetting 50 c.c. of the wine into a flask which has had a stream of carbon dioxide passed through it, adding 5 c.c. of sulphuric acid (1 : 3), and titrating at once with the N/50 iodine until the blue colour is permanent for a short time, after which the tannins, etc., in the wine begin to be oxidised. The difference between the total and the "free" sulphurous acid (*i.e.*, that existing as such and as alkaline sulphites), is termed "aldehyde" sulphurous acid by the author. By adding sulphurous acid to unsulphured wines, he showed that when the wine is kept the free sulphurous acid decreases and the aldehyde sulphurous acid increases, while the total sulphurous acid also decreases.

The author gives no summary as to the quantity of sulphurous acid in wines, but from the figures he quotes it would seem to average a little over 0.01 gram. per 100 c.c. of the Rhine wines which he analysed.

A. G. B.

REVIEWS.

THE PRINCIPLES OF THEORETICAL CHEMISTRY. By IRA REMSEN. Fourth Edition (London: Baillière, Tindall & Cox; 7s. 6d.)

The object of this work, as stated in the preface, is "to help students to get clear ideas in regard to the foundations of chemistry;" and, keeping this in view, the author has succeeded in producing a book which is not only extremely useful for the purpose for which it was originally intended but will be read with pleasure and profit by many who have long passed the period of studentship. The matter is placed before the reader in such a charming manner that he is unconsciously carried away with the subject, and experiences no tedium in the perusal of the work. Whilst in strict keeping with the present state of chemical theory, the author most sedulously distinguishes between the proven and the unproven, and carefully warns the reader against the prevalent error of over-theorising. The following statement is particularly apropos, and is one which many will cordially endorse;—"It cannot be denied that we are now in a period of chemistry which may fairly be called one of *formula worship*. More value is sometimes attached to a formula than to that which it is intended to represent. In consequence of this, it has happened that a large number of chemists have regarded the determination of a formula for a compound as the great object to be accomplished, and they have forgotten that what we ought to know, and what is of vastly greater importance to the science, is the chemical conduct of the compound." The fact that, within a comparatively short time, three editions of the work have been exhausted and a fourth demanded, shows that it must have attained a considerable amount of popularity. Since the appearance of the last American edition, Italian and German translations have appeared.

W. J. S.

UNTERSUCHUNGEN AUS DER PRAXIS DER GÄRUNGSINDUSTRIE. VOL. II.

By DR. EMIL CHR. HANSEN. (R. Oldenburg, Munich.)

This is a continuation of the account of Dr. Hansen's investigations in that difficult portion of the brewer's art which relates to the physiological processes concerned in fermentation, a branch of science in which this highly gifted author has so eminently distinguished himself.

The first chapter treats on the analysis of air and water for micro-organisms from a brewer's point of view. In this the author shows the futility of examining water by the method of Koch as used for hygienic purposes, since, as he points out, by far the larger portion of the organisms which develop on the gelatin plate are absolutely innocuous to either wort or beer. He prefers, therefore, to experiment directly on sterilised wort or beer.

In the second chapter, Pasteur's method of purifying yeast by cultivation in a wort acidified with tartaric acid is criticised at considerable length, and it is shown that, though this process readily frees yeast from bacteria, it favours the development of wild yeasts and suppresses the cultivated ones. Thus the gain on the one hand is counterbalanced by the loss on the other, and consequently it is impossible to obtain a pure yeast in this manner.

Chapter III. is devoted to an investigation of the diseases of beer caused by the *Saccharomycetes*. In it we find the interesting fact mentioned that Scheele was the first to apply practically the principle of sterilisation by heat. This he did in 1782, the substance operated on being vinegar, and the method he used does not differ materially from that in use at the present day. The author considers that it is in the large coolers that the wort principally receives its infection from the wild yeasts present in the air. He shows that the breeding ground of these organisms is the surface of fruits and berries in summer, and that in winter they lie dormant in the ground.

The fourth and last chapter gives a *resumé* of the extension of the author's system of pure yeast culture in recent years, and this has been very considerable. It is gradually coming into use in this country; and we learn that yeast grown from a single cell has been in use for some time past in one of our largest London breweries, where it has yielded the most satisfactory results.

A further development of the author's views has been applied to wine making. It is found that the flavour of the wine of a particular district depends on the variety of yeast found on the grapes of that district. Consequently, by fermenting the grape juice of any district with yeast from another district, the flavour of the wine is that of the district from whence the yeast was procured.

This process of selection is even applied to bacteria, pure cultures being used for souring the cream in cheese-making. The same principle is also applied to the fermentation of tobacco, for by selecting the bacteria different flavours can be produced at will.

W. J. S.

THE ANALYST.

APRIL, 1893.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held in the rooms of the Chemical Society, Burlington House, on March 1st, 1893. In the absence of the President, Dr. Dupré took the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed:—As Member, Mr. Walter Collingwood Williams, B.Sc. (*Honours in Chemistry and Physics, London*); as Associate, Mr. Hazlewood Irving Foster, assistant to Mr. James Baines, Hull.

Mr. W. Gathorne Hardy then read a paper on "A source of error in the Volumetric Estimation of Chlorides by Silver Nitrate Solution," which was followed by a discussion, and with this the Proceedings of the Society terminated.

ON THE USE OF CHRYSOTILE FIBRE IN PROXIMATE ORGANIC ANALYSIS.

BY THOMAS MACFARLANE, F.R.S.C., Chief Analyst, Inland Revenue Department, Ottawa, Canada.

(*Read at the Meeting, December 7th, 1892.*)

FIVE years ago I read a paper on Milk Analysis before the Royal Society of Canada, which is published in their Transactions for 1887. The method there described involved the use of asbestos, or rather chrysotile fibre as an absorbent, and this substance has since been found so useful in the examination of other substances, chiefly foods, in the Laboratory of the Inland Revenue Department at Ottawa, that I believe a description of the manner of applying it will be found interesting to the members of the Society of Public Analysts. Of course, asbestos properly so-called has been used for many purposes by chemists, and especially for forming the filtering layer in the Gooch crucible. The fibre so used is frequently called Italian asbestos, and belongs to the mineral species, hornblende or actinolite. It is not changed by ignition, and is not nearly so flexible as the fibre derived from chrysotile. Chrysotile is the fibrous variety of serpentine, loses 12 per cent. combined water on ignition, and is then easily rubbed to powder between the

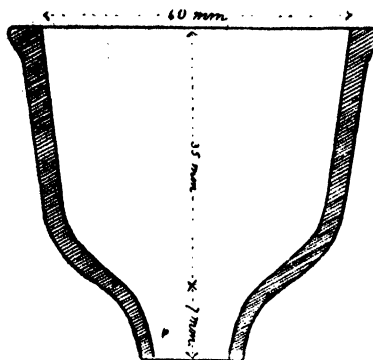
fingers. It is extensively mined in South Eastern Quebec, where it is found of a quality adapted for spinning and other finer manufacturing purposes. After being freed from rock and dust by grinding, sifting, and teasing, it assumes a woolly appearance; the fibres are silky and flexible, adhere to each other, and are quite destitute of the brittleness observed in glass wool, slag wool, or Italian asbestos. It cannot be called hygroscopic, for after long exposure to the atmosphere it contains only 0.57 per cent. moisture. The quantity used for an ordinary milk analysis is 2.5 grammes, which could only absorb by very long exposure 14 mgr. The change of weight during weighing must be therefore only infinitesimal. The chrysotile fibre is at present obtainable in London at the office of Messrs. Bolling and Lowe, 2, Laurence Pountney Hill, and it costs not more than 10d. per lb.

The use of chrysotile fibre as described in this paper is as a substitute for barytes, sand, powdered glass, calcined gypsum, &c., for absorbing and attenuating organic substances, so that their water may be conveniently removed and estimated, and so that the solvents used in their analysis may effectively permeate and act upon them. The advantage of the fibre is in serving this purpose in an eminent degree, while at the same time saving the chemist very much time, as well as trouble in manipulation. It is also to be remembered that the very complete drying effected by its means is of the greatest advantage in the operations to which the substance under examination may afterwards be subjected. I shall describe the manner of using it under the heads of the various substances, in the examination of which it has been successfully applied.

I.—MILK.

The chrysotile process of analysing milk has now been in use for over five years in the Inland Revenue Laboratory, as well as for a somewhat shorter time in that of the Department of Agriculture. It has been voluntarily adopted by all the public analysts in Canada, as well as by some in the United States. It has, of course, undergone slight modifications since first introduced, mostly for the purpose of saving time in treating a large number of samples. These are sometimes received at the rate of twenty or thirty a day in our laboratory at Ottawa, and I shall first describe the method as applied in this case. Samples are received from all parts of the Dominion, as far east as Sydney, N.S., and as far west as Regina in Assiniboia. They are always packed in ice, and arrive, as a rule, in good condition. Very likely, however, this packing in ice will be dispensed with in the near future, because on testing the method of preserving milk for analysis by introducing a small quantity of bichromate of potash, as proposed by Dr. J. E. Alén, of Gothenburg, I obtained very satisfactory results. This method will be found described in the *Deutsche Molkerei Zeitung* of 1892, pp. 224 and 416.

The glass tube used in the analysis, for containing the chrysotile fibre, has the shape and size shown in the figure. It is 40 mm. in diameter, 35 mm. in depth of bulb,



and 42 mm. in total depth to the end of the small tube. These tubes are made of well-annealed glass, and, after being numbered with a diamond and loosely filled with the chrysotile fibre, they are placed, to the number of 20 each, in copper trays or racks, measuring 9 inches by $7\frac{1}{4}$ inches, and having sides one inch deep, from which copper wires are stretched across, dividing the tray into 20 rectangular spaces. Four of these trays can be placed, one above another, in an ordinary box water bath, measuring $9\frac{1}{4}$ by $7\frac{1}{4}$ by 8 inches inside. The bath or oven we use is of

this size, contains 80 tubes, and is supplied with a Soxhlet metallic back-flow condenser, so that it can be kept at work for days continuously. Two extra trays have been found necessary when the samples are sent in rapidly, and as many as 120 tubes have been in use at one and the same time.

After having been thoroughly dried, a tray with its 20 tubes is removed from the bath, and the tubes placed in a desiccator to cool. Each tube is then weighed and replaced on the tray. This weight, which includes both tube and chrysotile contents, I prefer to take in grains for the purpose of saving calculation afterwards. The quantity of milk used for each analysis is then run into the tubes as they stand in the tray from a 10 c.c. pipette which delivers exactly or very nearly 10 grammes. Of course the point must be ascertained by experiment up to which the pipette must be filled in order to deliver this quantity. It is usually about three-eighths of an inch below the ordinary mark when the pipette is allowed ten seconds to drain. The sample is thoroughly mixed, and the pipette washed out with it before filling each tube with the 10 grammes. Each sample is usually assayed in duplicate, and in this way 20 tubes representing 10 samples are quite rapidly filled. The samples have usually consecutive numbers and so have the tubes as they stand in the racks from left to right. By adhering to this system there is little or no risk of interchanging the samples or tubes. The chrysotile fibre rapidly absorbs the milk, and if the tube has been properly packed with it nothing escapes at the lower end. In the case of sour milk there is, however, danger of this, and in this case a larger quantity of fibre must be used, and the curd mixed with it by gently stirring the assay with the point of the pipette.

After filling the 20 tubes the tray is replaced in the water bath, and left there during the night—six or seven hours at a temperature of 96° C. are usually sufficient, but I prefer to keep the bath running the whole night and to dry at a lower temperature, say as low as 85° C., by leaving the door open to a slight extent. In this way it is possible to dry the milk thoroughly without any discolouration. It is in thus drying at low temperatures and in the saving of manipulation that the advantage of the process consists. The fine fibres insert themselves between the particles of the milk and facilitate the

escape of the water. It is not necessary to stir or touch the assay after the introduction of the milk, as is required in cases where sand, gypsum, or barytes is used for the same purpose as the chrysotile.

After drying, the tubes are cooled in the desiccator and weighed. In doing this the assay is placed in one scale, the tare in grain weights above-mentioned in the other, and then gramme weights are added to the latter until equal poise is attained. This weight in grammes multiplied by ten gives the percentage of total solids contained in the sample. As a rule it is about 0.3 per cent. less than when the assay is performed in a platinum capsule. The solids and fibre together form a rather tough mass in the tubes and in this perforations are made with a sharp-pointed instrument in order to facilitate the subsequent extraction.

The removal of the butter-fat from the contents of the tubes is effected in a large chloride of calcium jar, 18 inches high, whose upper part has an inside diameter of $1\frac{7}{8}$ inches, without any contraction at the top. The tubes are placed one above another in this upper part, which is 14 or 15 inches long, and capable of containing 12 or 13 of the assays. The tubulus in the lower part of the jar is then tightly corked, and the jar filled to above the level of the highest assay with petroleum ether, having a specific gravity of about 0.670, with boiling point ranging from 60 deg. to 85 deg. C. The jar is then closed tightly with a bung, and the whole set aside for two hours, during which time the assays are thoroughly permeated with the petroleum ether and most of the butter fat dissolved. The ether is then withdrawn by the tubulus at the bottom of the jar and the tubes allowed to drain. Fresh ether is then poured in upon the uppermost tube, when it percolates through all the other assays and collects in the receptacle at the bottom. After the latter has been filled four successive times by the ether thus used to wash away the butter fat, and all the ether has drained away from the tubes, the latter are freed still further from ether by drawing a current of air through them by means of the ordinary apparatus used for quick filtering. The tubes are then exposed in a steam bath to a temperature of 95 deg. or 100 deg., which completely frees them from the ether. They are then cooled in the desiccator and weighed, the loss being the butter fat. A much larger quantity of petroleum ether is used in this way than in working with the Soxhlet and other extracting apparatus. In our laboratory, however, a copper still heated by steam is used for recovering the ether, and the distillation of the extra quantity used occasions very little additional trouble.

Of course, the Soxhlet apparatus may be used as described in my paper of 1887, and much of our work has been done in this way. We have also made use of a Stutzer tube similar to that described in Böckman's "*Untersuchungs Methoden*" (edition 1884, page 579). Instead, however, of supporting the inside tube by means of a spiral wire, the Stutzer itself has three protuberances jutting inwards at its contracted part upon which the tubes containing the fibre and milk rest during the extraction. In such a Stutzer as

many as five or six tubes may be operated on at the same time. In an interesting paper read before the Royal Society of Canada in May, 1890, Mr. Frank T. Shutt, chemist of the Central Experimental Farm, has shown that the extraction of the butter fat is performed at least as effectually in these Stutzer tubes, and, in fact, his figures show that the Stutzers give slightly better results than when the extraction is performed in Soxhlets.

The analysis of milk in these tubes may be carried further than the determination of the butter fat, although the amount of the latter and the total solids is all that is usually required. The tube and its contents may be extracted with alcohol, and from the loss in weight the percentage of lactose ascertained. In this case, however, it is necessary to deduct from the result the weight of a small quantity of coagulum which separates from the alcoholic solution, and the ash yielded on evaporating the latter and igniting the product.

II.—BUTTER.

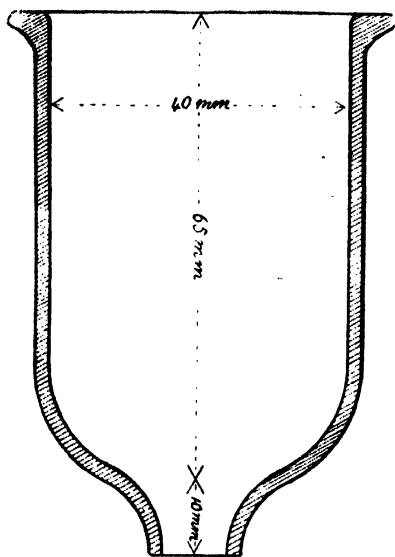
The application of the chrysotile fibre to the examination of butter concerns only the estimation of the water, fat, salt, and curd which it contains, and has no reference to the detection of foreign fats. Where the tubes or cups are to be used for butter the chrysotile fibre is placed in them in successive layers, so that the top one may be removed without disturbing the others. After a tube is dried and weighed, the top layer of fibre is removed, the butter to be examined introduced, and the top layer replaced carefully, so as to prevent any of its constituents from being removed by a possible splashing of the solvents. On weighing the tubes again the amount of butter taken for one assay becomes known. When the tube is placed in the water-bath, the butter melts, is absorbed by the fibre and the water is dissipated in the course of an hour. On cooling and weighing the tube, its amount is ascertained from the loss. By means of petroleum ether the fat is next dissolved out, and its amount obtained in the same way. Water next removes the salt, the curd is left insoluble, and from the next weighing the amount of both ascertained.

In heating the sample for an hour or two in the fibre in order to the dissipation of the water, there is not much reason to dread error from the oxydation of the butter fat. A sample exposed for seventeen hours in the water-bath, at a temperature of 90°C, only experienced an increase in weight of 0.82 per cent. calculated on the butter fat.

III.—CHEESE.

The glass tube or tubulated cup which is used for the examination of cheese is of larger dimensions than that used for milk analysis. It has an outside diameter of 40mm., and a greatest length of 75mm., the depth of the wide part being 65mm. In packing it with the fibre it has been found advantageous to place a small piece of platinum or wire gauze over the small opening in the bottom to prevent the fibre from being drawn down into it when the contents are being subjected to the action of the filter pump. The fibre itself is introduced in successive loose layers, so that they may be removed and replaced singly when desired. After the tube with its contents has been dried and weighed, all

the chrysotile, excepting the layer at the bottom, is removed, and 5 grammes of the cheese in thin slices mixed or kneaded up with it, replaced in the tube and then covered over with the uppermost layer of the fibre. On weighing the whole, the weight of the cheese employed may also be again ascertained. The tube is then dried in the water-bath at a temperature of 98° , and a very perfect elimination of the water effected. This is proved by the close agreement of duplicate analyses. On cooling and weighing the tubes the loss in weight indicates the quantity of moisture present.



The tube is next subjected to extraction in a Soxhlet or Stutzer, and the loss which it experiences regarded as butter-fat.

The contents of the tube are next subjected to the action of warm water containing one per cent. of acetic acid. It is, in fact, washed therewith, while the tube is connected with the vacuum pump. The loss thus sustained is lactose, salt and soluble matter, the latter including, of course, the peptones

and other soluble transformation, products of the caseine which have been produced during the ripening of the cheese.

That part of the cheese remaining as residue among the chrysotile fibre in the tube is regarded as unaltered casein.

IV. -- FATS AND OILS.

The amount of water contained in these, may, of course, be ascertained in exactly the same manner as described under butter, and this plan has been very successfully carried out in our laboratories in the examination of lards, some of which have been found to be adulterated with 5 to 25 per cent. of mechanically combined water. In the course of these analyses it was found that some lards, although heated for only a short time, increased in weight. Following up this observation, it was found that such lards were generally free from water, but adulterated with cotton seed oil. In fact, while pure lards exposed to a temperature of 95° C. for eighteen hours in chrysotile fibre seldom show a greater increase than 2.5 per cent., compound or adulterated lards always show over 3 per cent., and not unfrequently as high as 4.5. It is not supposed that this test would, taken by itself, be sufficient to condemn a lard or otherwise, but in conjunction with other data it has been found valuable. In cases where by a judicious admixture of beef tallow, lard, and cotton seed oil, the lard shows a satisfactory iodine number, it has been found possible to detect such adulteration by the use of the nitrate of silver test, in conjunction with the one now referred to.

It is quite possible that this method of determining the capacity which oils and fat have of absorbing oxygen may be found worthy of a wider application. I may mention the following percentages of increase in weight, which the following substances experienced on being heated for 18 hours in chrysotile fibre:—

Cotton seed oil	5.45—5.64	per cent.
Oleo oil from Chicago	0.37—0.44	„
Oleo stearine	no increase	
Beef tallow, home rendered and filtered,	no increase				
Mutton tallow	0.21—0.31	„
Porpoise oil	4.85—4.98	„
Neats foot oil	3.06—3.29	„
Olive oil (Barton and Guestier)	4.29—4.49	„
Linseed oil	4.72—5.04	„

Of course, these absorptions vary as the time of exposure is increased or otherwise. In the case of linseed oil the increase is much greater in the cold when plenty of time is allowed. Some very interesting results are obtained when oils are in this way allowed to attain the maximum degree of oxidation, and are then subjected to the action of various solvents, but these results are not yet complete in my hands, and should, at any rate, be treated of in a separate paper.

V.—SOAP.

It must have occurred to some of the members now present that the same method of drying which I have described as applicable to cheese would also answer for soap, and such is the case. In several respects the use of chrysotile fibre is preferable to that of sand as described by Mr. Wilson in the *Chemical News* of 21st October last, p. 200. The soap may either be introduced into the fibre in the same manner as the cheese or after solution in alcohol. After drying at 98° C. closely agreeing results are obtained with duplicate samples.

The dried soap entangled among the fibre in the tubulated cup is then subjected to successive extractions, in Soxhlets or Stutzers, by the following solvents: petroleum ether, alcohol and water, the loss sustained by each treatment being noted. The first loss represents unsaponified fat; the second soaps, free alkali and glycerine; the third alkaline carbonates, salt, &c., while the insoluble substances, whether of organic or mineral origin are ascertained from what remains in excess of the original weight of the tube and fibre.

Of course the ethereal, alcoholic and aqueous solutions obtained in this way are all available for other necessary separations and determinations.

VI.—SPIRITS AND MALT LIQUORS.

It is chiefly in the determination of the amount of extract contained in these that the chrysotile has found application. The result has been a more perfect elimination of the

water, and a decrease in the percentage of the extract. The following statement shows the differences between this and the ordinary method of determining the extract in such beverages :—

	Grammes Residue per litre when dried at 100° C.		Percentage of additional loss when dried in chrysotile fibre.
	In Platinum Capsule.	In Chrysotile Fibre.	
Champagne Cognac, 1865	16·654	14·746	11·46
Hennessy's Cognac V.O.	9·180	8·520	7·19
Gooderham & Wort's Rye Whisky ...	1·959	1·842	5·97
Canadian Porter, Walkerville B. Co.	55·200	53·400	3·26
Allsopp's Red Hand Ale	56·900	55·475	2·51
Brading's Ale, Ottawa	37·050	36·650	1·08

With reference to the amount of extract contained in worts and de-alcoholised beer, it is admitted by most authorities that the percentages given in the Schultz tables as corresponding to certain specific gravities are too high. Schultz obtained his figures by determining the specific gravity of various worts at 15° C. and then evaporating to apparent dryness at from 75° to 80° C. In this way it is not possible to obtain an extract free from water, and recognising this, Dr. A. Elion constructed in April, 1890, a new set of tables (*Zeitschrift für angewandte Chemie*, 1890, p. 291) based upon experiments in which the extract was determined by drying in a rarefied atmosphere at 97° C. These values are much lower than those of Schultz, but it is not certain that even they show the amount of dry substance perfectly free from water. Our method of drying in chrysotile at 92° C. gives even lower results and in the following table are given some of those obtained in a series of determinations of de-alcoholised beers as also, for the sake of comparison, the specific gravities and the amount of extract corresponding thereto according to Schultz, to Elion, and to the saccharometer tables of Bates :—

No. of Sample.	Description of Sample.	Sp. gr. of spent beer 16° C.	Grammes solid in 100 c.c. according to			Grammes solid in 100 c.c. dried in chrysotile fibre.
			Schultz.	Elion.	Bates.	
4.	Sparkling Amber Ale	1·02191	5·79	5·40	5·55	5·413
9.	Hamilton's London Amber Ale ...	1·01194	3·11	2·96	3·06	2·801
10.	Dörbecker's Lager Salem	1·02044	5·41	5·09	5·06	4·070
16.	Dow's India Ale	1·02148	5·69	5·36	5·31	4·925
18.	Toronto Brewing Co. India Pale Ale	1·01190	3·11	2·96	3·06	2·625
20.	Carling's India Pale Ale	1·01709	4·50	4·26	4·31	3·940

From these figures it will be observed that as stated above, the results obtained by drying in chrysotile fibre are invariably lower than even Elion's figures, although in some cases the difference is very slight. Elion's experiments were made with genuine malt extracts and beers, and it is still uncertain as to whether these differences are owing to the method of drying, or to something abnormal in the beers above mentioned.

In order to ascertain the nature of the constituents of these beer extracts, the tubes containing them and the fibre are subjected to extraction by alcohol of 96 per cent. (by volume). Sugar which has escaped fermentation and other substances are thus removed, and the tubes, after drying, &c., are weighed. The substances remaining in the chrysotile fibre are albuminoids and dextrin. A nitrogen determination on the original beer by the Kjeldahl method enables the quantity of the former to be calculated, and on its subtraction from the amount of residue the quantity of gum and dextrin becomes known. This is an important figure which renders it possible, in conjunction with the degree of fermentation (Vergärungs grad., *i.e.*, the percentage loss of original constituents in the wort during fermentation), to determine whether saccharine substances have been used in the brewing.

VII.—SYRUPS AND MOLASSES.

The difficulty of drying these products is well known, but it is overcome by the use of chrysotile fibre. The quantity used for analysis must not, however, be introduced direct among the fibre. It must be diluted with four or five times its weight of water, and then an aliquot part of the solution may be pipetted into the tube among the fibre. After drying these for 48 hours, there is no difficulty in obtaining concordant results.

The tubes thus dried may be extracted with alcohol and the solution used for the saccharometer. I believe more correct results are thus obtained than by direct polarisation; but to discuss this matter would go beyond the limits of this paper.

VIII.—COFFEE.

Not only has it been found possible to treat with advantage liquids, oils, and viscous substances in chrysotile fibre, but it has also been used for enveloping and enclosing powdered substances, in place of the usual capsules of filtering paper, while they are being acted on or extracted by various solvents. One of the most important of these is coffee, regarding which I shall venture to go more into detail. The tube in which it is treated is the same as that above-described under the heading "Cheese." The manner of filling it with the fibre is the same, and care must, of course, be taken, in this, as in other cases, that it is well teased out and not packed too closely in the tube. Owing to the large number of samples which are usually delivered to the laboratory at one time, the tubes are arranged in copper racks holding 20, all properly numbered, and are dried for 24 hours in the water oven at 100 degs. C. The tubes are weighed from the desiccator and immediately returned to it. After their weight is known they are each charged with from 5 to 7 grammes of the coffee to be analysed, which has been previously ground to a standard fineness. The filling is performed by rapidly removing the chrysotile layers with metal pliers, over black glazed paper, and replacing them with a layer of, say, 3 or 4 mm. of coffee between each. A very thick plug or layer of fibre is placed on the top, and the loaded tube is immediately weighed to ascertain the exact amount of the sample taken.

After 24 hours in the water oven at 100 degs. C., the weight is again taken, and the loss is stated as moisture.

Curiously enough, this loss is somewhat greater than when the coffee is dried on thin layers on watch glasses. In 18 samples dried by both methods simultaneously, the following numbers were obtained:—

		Loss at 100 degs. C. in 18 hours.	
Sample,		On Watch Glasses,	In Chrysotile Fibre.
1.	...	6.73	7.07
2.	...	6.13	6.44
3.	...	4.20	4.51
4.	...	5.65	6.11
5.	...	5.28	5.58
6.	...	9.84	9.80
7.	...	10.39	10.69
8.	..	3.83	4.70
9.	...	5.27	5.65
10.	...	5.71	6.01
11.	...	5.12	5.32
12.	...	4.74	5.01
13.	...	6.12	6.42
14.	...	5.39	5.64
15.	...	4.08	4.51
16.	...	6.80	7.14
17.	...	4.78	5.08
18.	...	4.86	5.04
Mean		5.82	6.15

The above are samples of commercial coffee, some of them very much adulterated. The loss of weight is practically constant at 18 to 20 hours. In 20 tubes, containing an average of 6.133 grammes each, the loss of weight in 20 hours averaged 262 milligrammes, and for the next 24 hours a further loss of only 3.9 milligrammes occurred.

The tubes are now exhausted by petroleum ether, which may be done either in Soxhlet or Stutzer tubes, two or three of the assays being treated at once. A very large number of samples have been worked over in this way in duplicate, with very satisfactory results. Genuine coffees yield from 10 to 12 per cent. of fat by this treatment. One genuine sample gave only 8.20 per cent., but, in this respect, stands alone among nearly 50 pure coffees. No sample gave as much as 13 per cent., although 12.5 has been reached in a few instances. Several of the district analysts have obtained higher figures, but this is owing to the use of ethylic ether in the extraction,

instead of petroleum ether. The quality of the latter used in the Inland Revenue Laboratory is obtained by distillation from "deodorised gasoline" at temperatures ranging from 60° to 85° C. The percentage of fat contained in adulterated coffee varies from 12 to 2 per cent., and as a general rule it may be assumed that the lower the percentage of fat the greater is the amount of adulterants in the coffee. It would, however, be unsafe to depend entirely on this, as there is nothing to prevent the manufacturer from introducing foreign fat into the mixture if he finds it to his interest to do so. Chicory gives about 1 per cent. of extractive matter to petroleum ether, and it may be added here that three samples of roasted barley yielded respectively 1.31, 1.54, and 1.42 per cent. to the same solvent. All the percentages of fat extracted are calculated on the dry substance.

The tubes are next treated with boiling water in order to determine the amount of water extract. They are placed in the rack and boiling water poured into them, and after standing about 15 minutes they are placed on the jars connected with the filter pump, in the same manner as for the washing by ether, and repeatedly washed with boiling water until the washings have but little colour. This requires an average of 10 minutes for each tube, and about 250 c.c. of water. On being dried as thoroughly as possible by the pump, they are returned to the oven and kept at a temperature of 100° C. for 48 hours. The loss of weight is the water extract, and amounts to about 22 per cent. in the case of genuine coffee. Chicory gives nearly 80 per cent. (77.73 per cent. according to our experiments), and it has been proposed to make use of the percentage of water extract obtained in this way as a substitute for the indications of the ten per cent. decoction in the cases of mixtures of coffee with chicory only. In the case of samples containing other adulterants as well, the amount of water extract varies from 25 to 68 per cent., the high figures being occasioned by the sugar, caramel, gum, soluble starch, and dextrin contained in the substances which are mixed with the coffee in order to produce a cheap article.

The foregoing descriptions do not by any means exhaust the list of substances whose examination or analysis has been facilitated by the use of chrysotile fibre and the various glass tubes mentioned. Mustard may be treated in exactly the same manner as coffee, and its moisture and fixed oil contents conveniently determined. Pepper may also be distributed among the fibre, and the moisture and alcoholic extract determined. I have also introduced common gunpowder among the fibre in these tubes, and by successive extractions with carbon disulphide and water determined its percentages of sulphur, nitre, and carbon. The examination of varnishes is much facilitated by driving off the volatile constituents in these tubes and then subjecting the residue to the action of various solvents. Mr. Shutt informs me that he has conveniently and successfully performed the analysis of condensed milk by using this system, and I have not the slightest doubt that the water contained in honey could be easily eliminated and estimated by the use of chrysotile fibre. It has also seemed to me that it, in conjunction with the tubes for containing it, constitutes

the best possible means for carrying out to a considerable extent the plans described by Dragendorff and others in the analyses of plants. I sincerely trust that my fellow members of the Society of Public Analysts will give these methods and suggestions some consideration, for I feel convinced that their adoption in whole or in part will materially lighten their labours and economise their time.

Although I have to bear the chief responsibility of introducing the use of chrysotile fibre into the methods of the Inland Revenue Laboratory at Ottawa, I must not neglect to state that much of the work referred to in this paper has been performed by my assistants, Mr. A. McGill, M.A., Mr. F. W. Babington, F.I.C., and Mr. A. L. Tourchot; and I take this opportunity of gratefully acknowledging the interest, zeal, and ability with which they have worked out some of these problems in proximate organic analysis.

DISCUSSION.

The President said that Mr. Macfarlane had dealt with a great variety of subjects in his paper. It was only quite recently that Mr. Richmond had again brought before the Society the system of evaporating milk by means of asbestos wool, which he had been experimenting with. No doubt it was unfortunate that public analysts, something like fifteen years ago, at the instance of Mr. Wanklyn, had ceased to use the method which was until then universal, of evaporating milk with diluting substances like sand or pumice. He was surprised at one or two remarks made by Mr. Macfarlane. He (the President) would have expected that with the use of the chrysotile fibre he would have obtained higher total solids than without it, for the reason that, if such a substance as milk, or anything of a sugary nature were dried *en bloc* in a platinum basin, it was excessively difficult to dry, in fact it was almost impossible. Milk, when evaporated, always got more or less discoloured, whilst Mr. Richmond did not find any such discolouration by the use of asbestos fibre for evaporating milk, and he (Mr. Richmond) obtained high results. In the case of wine and malt wort, the figures for total solids obtained by evaporation, as proved by Dr. Dupré a good many years ago in the case of wine, were always smaller than those obtained by the specific gravity of the de-alcoholised liquid. Dr. Dupré had pointed out that a certain amount of dehydration took place, which altered the character of the residue. The observation as to the oxygen absorbed by oils would be a very great help, especially in testing for cotton seed oil. Cotton seed oil was now so treated, with a view to deceiving analysts, that the old silver test sometimes appeared to fail. He had had a number of samples of lard which he was almost convinced contained cotton-seed oil, or some very similar oil. If a corroborative test could be found, it would be exceedingly useful. At the same time the oxygen absorption test for oils had been used for a great many years in examining many forms of oils.

Mr. H. Droop Richmond said he would like to state, in reference to the President's remark, that he had not claimed the asbestos method as his own. In his paper (ANALYST

xvii., 225) he had mentioned that some years ago the asbestos method had been introduced in America by Mr. Macfarlane or Mr. Babcock. Would Mr. Macfarlane tell the meeting whose method was first published.

Mr. Macfarlane replied that there could be no doubt that Mr. Babcock first introduced the use of asbestos, but he thought that Mr. Babcock did not introduce the use of crysotile fibre or the method of obtaining the results by difference. He did not bring forward this method as anything particularly new, except in the manner of applying it so that the butter fat could be determined by difference, and a much larger number of samples analysed than the analyst otherwise could deal with in the same time.

Mr. Richmond wished to ask Mr. Macfarlane how the results for fat agreed with the ordinary methods; that is to say, with the extraction of the fat by the Adams or Werner-Schmid processes. He (Mr. Richmond) wished to say that he shared the President's surprise at the results of Mr. Macfarlane's determinations in the case of milk, being below those given by the ordinary method. The results by the asbestos process he had found, if anything, a little higher; and on drying for a very considerable number of hours—up to, in some cases, close on 60—he had found that the weight remained practically constant; sometimes it had gone half a milligram up or down. He could not get it to lose any more. He (Mr. Richmond) put the asbestos in a platinum basin and ignited it, and all the water in the asbestos, whether it was condensed moisture or whether it was combined water, was driven off. Mr. Macfarlane had stated that he sometimes had his milk samples sent him in ice. He (Mr. Richmond) was strongly of opinion that it was a bad thing to put milks in ice. It caused separation of fat in such a way as to render proper admixture afterwards difficult. He preferred to have his samples kept at a warmer temperature, even at the risk of their curdling sooner.

Mr. Macfarlane did not think that this method had been compared with any other than that of ordinary evaporation in platinum vessels, and certainly the loss was greater in crysotile fibre than it was in platinum.

The President asked whether the total solids were determined by drying to constant weight, or by the usual process of drying for three hours?

Mr. Macfarlane said that it was possible to dry the milk in four or five hours at a temperature of 98° C., but he preferred to leave the samples in the water-bath over night at about 92° C. In determining the solids in platinum, and comparing the results with those obtained in crysotile fibre, the same time and temperature was employed. There could not be any doubt that more water was eliminated by using crysotile fibre. As to any loss the crysotile fibre might sustain when dry, it had been exposed to temperatures under and up to 100°C. without its losing weight. This variety of crysotile loses 12 per cent. combined water on ignition. In this case it loses its character altogether, and falls to powder. With regard to Mr. Richmond's question, no other plan could be devised

than packing it in ice ; unless, indeed, the method of Dr. Alén, of Gothenburg, by bichromate of potash, should be found to answer, when the milk had to be sent long distances. When the milk arrived cold enough he had never had any difficulty in consequence of the separation of the cream.

Mr. Richmond wished to ask Mr. Macfarlane how the results under this method compared with the Adams and other processes ?

Mr. Macfarlane did not think that he had made such comparisons. He did not use the Adams method. There was so much manipulation connected with it that the large number of milk analyses required could not possibly be got through in the proper time. He had to adopt some other method whereby the manipulation was not so great. The actual variation had been determined by Dr. Ellis, of Toronto, between this and the Adams method, with the practical result of his adopting it instead of the Adams ; not that it was considered more correct, but because a large amount of time and trouble was saved. A good deal of work had also been done by Mr. Schutt, of the Experimental Farm Laboratory at Ottawa, in these comparisons, and he (Mr. Schutt) had always found a less quantity of total solids by using the fibre than otherwise.

Dr. Muter said that as time rolled on they returned to the old forgotten methods. Mr. Macfarlane's process was practically similar to the one that he (Dr. Muter) practised twenty-seven years ago, when he first made a speciality of the analysis of food and drugs. It was a very good method, because it gave the actually dry residue ; and was only abandoned in deference to the general agreement to adopt the Wanklyn method, as to which he agreed with the President's remarks. In the old way a small funnel, with a glass cover, was plugged, nearly filled with sand, dried in a specially constructed bath, and weighed. The milk was run on to the sand, and the whole again dried and weighed. The funnel and its contents were then percolated with ether, and the fat thus removed weighed. The residue was then percolated with warm dilute spirit, to remove sugar (estimated by Fehling) ; and finally the sand was dried and submitted to combustion with soda lime for the proteids. Then nothing was known as to the constancy of the solids-not-fat, and consequently full analyses were always made. He merely mentioned this as a matter of curiosity, but even now it was not a bad way of attacking condensed milks, which contained an abundance of cane sugar. As a rule, with the sand, or other similar method, the total solids came 0.1 to 0.2 below those taken by simply drying in a dish. For ordinary commercial work he considered that no process was so good as the solids, gravity, and Richmond's slide rule.

Mr. Macfarlane in compliance with a request made by the President, then gave some particulars as to the labours of Public Analysts in Canada. The laboratory branch of the Inland Revenue Department was charged with the administration, not only of the Adulteration Act, but of the Fertiliser's Act, and he would try to state how this was carried out. There were eight district analysts in the Dominion, one in each of the cities

of Halifax, St. John, Quebec, Montreal, Ottawa, Toronto, London, and Winnipeg. Those gentlemen were professional men, of course, and prepared to do all the analytical work the public had to offer them. They were also appointed public analysts by the Government, and in order that they might take their part in carrying out the Adulteration Act, they were supplied from time to time in the course of the year with a certain number of samples to analyse. These samples were collected by food inspectors, of whom there were as many as there were analysts, that is one for each district. The instructions received by the food inspectors, were issued by the laboratory branch after the Commissioner of Inland Revenue having been consulted as to what articles should be analysed. Orders were issued to the food inspectors to collect so many samples of foods, drugs, or agricultural fertilisers, some perhaps of a particular kind, and to deliver them to the public analysts. The food inspectors went into the shops and places of business and asked, as if they were ordinary purchasers, for the articles in question. According to the law, and according to the practice in Canada the vendor was then and there, after the purchase had been made, informed of the purpose for which the sample was obtained. The sample was then divided into three parts, each sealed up, one left with the vendor, one sent to the district analyst, and the remaining one to the departmental laboratory at Ottawa. The next step was the analysis by the district analyst of the sample and his report to the Commissioner of Inland Revenue. The analyst was paid certain fees on the delivery of his certificate showing whether it was or was not adulterated. The certificates were then inspected by himself (Mr. Macfarlane), and the results placed on record in the books kept by the branch at Ottawa which were properly systematised. He was able with the aid of the laboratory staff, to go through all the samples which had been analysed by the district analysts and check their results, but as a rule only those found by them to be adulterated were considered to be worth revision in this way. In such cases the sample sent to Ottawa was analysed in the laboratory there, and if the results obtained by the local analyst were confirmed, the next proceeding was to "apply the Act," carrying out its provisions in proper order. Of course they could at once report certain cases to the Department of Justice for prosecution, but the Act gave them the power of previously intimating to the vendor that his sample had been found to be adulterated: and they had also the power under the Act of calling upon him to pay the cost of collecting and analysing the sample, which cost varied from eight to sixteen dollars. The vendor could elect to pay the fine, or to refuse and stand a prosecution. This plan was adopted, and an amendment of the Act was inserted to that effect, because it was found that in a great many cases, if the vendor were prosecuted, and a conviction was obtained, the resulting fines were far from adequate to pay the expenses of the prosecutions. The consequence was that the grant of money for carrying out the Act which was given by the legislature, had to stand large outlays for legal expenses. On this representation being made to the Minister of Inland Revenue he suggested the method now in force for overcoming the difficulty. In three-

fourths of the cases the fines were paid without any prosecution being necessary, the vendor preferring to pay, rather than to be subjected to a prosecution. The names of those who did not pay, were given in to the Department of Justice, and a prosecution took place in the ordinary way. In law there, as here, there was much uncertainty, but in the majority of cases convictions were obtained. He could only remember that they had failed in the case of baking powder, the lawyers raising the question as to whether that was, or was not a food. In addition to such legal proceedings, bulletins were published in which the names and addresses of persons from whom samples had been taken were given, and also the results of the examinations, both of the genuine and adulterated articles. The publication of these bulletins was quite anxiously looked for by the public, and the Press readily took notice of them. The local newspaper, too, was sure to publish the names of those individuals in their neighbourhood who had been found to sell adulterated goods; so that, on the whole, during the last six years it could fairly be said that the branch had done a great deal of good, and had done much in the direction of improving the quality of the articles sold throughout the whole Dominion. They also had the support of the public, and, to a very large extent, of the dealers also, as well as the manufacturers. It was merely a question of getting the Act applied to a sufficient extent. The difficulty was that, notwithstanding, a good many adulterators escaped. If the Act were administered in its entirety, and all of its stringent provisions applied, the officers of the branch might possibly make nuisances of themselves. Their powers were so great that they might end by bothering everybody. Care had to be taken that unreasonable prosecutions were not instituted, and as long as this was done they would have the support of the public and the traders. The bulletins before the meeting showed the way in which the names were published, and it would be seen how each article was treated.

The President asked Mr. Macfarlane what he considered reasonable limits with regard to milk. What course did he adopt with those samples which gave results only very slightly below the average?

Mr. Macfarlane replied that such cases were left severely alone. They certainly would prosecute in every case where the fat was less than 3 per cent.—even slightly less; anything below 3 per cent. would be condemned. The bulletins showed the judgments that had been given. He did not think he could at the moment pick out the doubtful samples which had been referred to by the President. He had sometimes cases of comparatively rich milk which, he was tolerably certain, had been watered; but as there were plenty of worse cases to be attended to, those were put down as “below average.” It had been found by the analysis of a great number of samples of genuine milk that the average percentage of fat in Canadian milk amounted to 3.75. Perhaps not more than 1 in 100 would be found to contain less than 3.45 per cent. of fat. They would be called “under average,” although they were genuine. They (the analysts) were perfectly justified in

calling them "below average." What he had proposed was that the lowest limit should be 12 per cent. of total solids, of which 3.5 should be milk fat, and 8.5 other solids. No standard had been established by the Government in connection with this recommendation. The Act gave the Government the power to fix the limits of variability in the case of any article, but they were unwilling to do so unless there was a very great necessity for it. He did not think that his branch was impeded in its operations because there was no legal standard. Any milk containing less than 8.5 per cent. of non-fatty solids, or any milk containing less than 3 per cent. of fat, was condemned, and a prosecution was instituted, with a very good chance of obtaining a conviction.

Mr. Cassal asked Mr. Macfarlane whether he would calculate the percentage of fat abstracted on a limit of 3.5 per cent. of fat, or of 3.0 per cent. of fat.

Mr. Macfarlane stated that the adulteration branch would not prosecute for anything between 3.0 and 3.5, but that it would do so if the sample contained less than 3 per cent.

Mr. Cassal explained that he wished to know whether Mr. Macfarlane would report that a particular sample of "milk" deficient in fat had had a certain percentage of its original fat abstracted, and, if so, whether he would calculate that percentage on the limit of 3.5 per cent. of original fat, or of 3 per cent. He gathered that Mr. Macfarlane reported analytical data, namely, percentages of solids, fat, and so forth in his certificates, or in the bulletins, a course which he (Mr. Cassal) was glad to say had now been given up by most of the leading English public analysts; but he supposed that when cases of adulteration went into Court the meaning of the analytical data was given in the form of percentages of added water or of fat abstracted.

Mr. Macfarlane stated that it was not necessary to state the solids, fat, &c., he only went as far as was necessary to obtain a conviction.

The President wished to know, supposing Mr. Macfarlane, as head of the department, had analysed a sample which the district analyst had reported as adulterated; and supposing that the two results happened to differ, whose opinion would prevail? There were two ways of differing—either through the existence of an error, on one side or the other, or through a difference in the opinion founded on the analytical results. One chemist might arrive at the same analytical results as another, but he might draw different conclusions and give a different opinion. A case had occurred quite recently in London where a sample of milk had been analysed by three persons. The first said the milk was skimmed; the second said that it was both skimmed and watered; and the third said that the sample might have been genuine; while the results obtained by each did not differ very widely. If such a case occurred in Canada, how would it be dealt with, and what would be the consequences?

Mr. Macfarlane could only conceive of such cases occurring in the following

manner. As he had stated, the vendor retained part of the sample sealed up, and he was at liberty to employ any chemist to examine that sample, but during the whole of the six years that he (Mr. Macfarlane) had been in charge of the branch, he had not known a case where this sample had been referred to a private analyst. The vendors could refer them to the chief analyst, but they did not, as a rule, go to that trouble. In a few cases the vendors had sent their samples to Ottawa, and in one case his department found that an interchange of samples had taken place. When the vendors referred their samples to headquarters, they usually accepted the result without question. In the event of there being any difference of opinion between the chemists at Ottawa and the district analyst, it was usually found that by consulting together differences could be explained, No pressure was used by the department. The district analysts were perfectly independent; but it was quite within the province of the chief analyst in any particular case to suggest that the opinion of a district analyst was not quite correct, and to specify his reasons for the suggestion, and there were instances in which the certificates had been modified accordingly. It was not thought necessary that such differences of opinion should be made public; the whole matter was arranged privately. Everything was done to prevent injustice to the traders of the Dominion; but at the same time when prosecutions became necessary the analysts were perfectly united. It never happened that they gave opposing opinions in court; they would not allow such to become public. If differences existed which could not be reconciled, the matter was dropped, and no prosecution was attempted.

On the Determination of Ferric Oxide and Alumina in Mineral Phosphates
G. Mariani and E. Tasselli. (*Staz. Sper. Ag. Ital.*, xxiii., 31.)—The authors show that Gläser's alcohol method has the following defects:—

- (i.) The precipitation of a small quantity of calcium phosphate with the ferric and aluminium phosphates.
- (ii.) The possible precipitation of basic phosphates, if all the iron and alumina are not in the mineral combined with phosphoric acid.
- (iii.) The partial solubility of ferric and aluminium phosphates in dilute acetic acid.
- (iv.) The decomposition of ferric ortho-phosphate by boiling water into soluble acid phosphate and insoluble basic salt.

They propose the following method:—1 to 5 grams of the phosphate are boiled in a flask with 15 c.c. strong hydrochloric acid for 10 minutes, and are then diluted with twice the volume of water; a few crystals of potassium chlorate, and several drops of nitric acid, and the liquid boiled to drive off the excess of chlorine. This is filtered, the filtrate and washings measuring about 150 c.c.; to the cold liquid a solution containing half a gram of ammonium phosphate is added, 2 c.c. of glacial acetic acid, and then

dilute ammonia drop by drop, until a small permanent precipitate remains; 2 c.c. of acetic acid are then added, and the liquid, after repeated shaking, is left for a couple of hours. The precipitate is collected on a filter, and washed with 1 per cent. ammonium phosphate solution.

The funnel is placed over the flask in which precipitation took place, and the precipitate is dissolved by a minimum of hydrochloric acid (sp. gr. 1.12), and a second precipitation is performed as before. The precipitate is well washed with the ammonium phosphate solution mentioned above, the filter dried, and burnt at a dull-red heat.

Half the weight of the residue is taken as ferric oxide and alumina.

A Thomas slag, two mineral phosphates, and an artificial mixture containing 1.60 per cent. of ferric oxide and alumina were analysed.

	New Method.	Greatest difference of duplicates.	Gläser's Method.	Greatest difference of duplicates.
Thomas slag	19.12	0.31	19.48	0.30
Mineral phosphate ...	4.01	0.45	3.72	0.25
" " ...	1.88	0.15	1.76	0.21
Artificial mixture ...	1.574	0.040	1.514	0.100

The authors do not consider their method perfect, but they put it forward as having many advantages.

H. D. R.

Some Points in the Determination of the Intensity of the Colour of Wines, and in the Determination of Free Tartaric Acid. L. Sostegni. (*Staz. Sper. Ag. Ital.*, xxiii., 10.)—The author shows that when comparing the colour of wines with that of a standard solution of fuchsine (0.05 grs. per litre) the results vary very largely according to the length of the column used in the comparison. Duboscq's colorimeter was used. He also shows that different specimens of fuchsine have very different intensities of colour.

From his experiments on malic acid, he concludes that acid potassium malate is precipitated along with acid potassium tartrate in alcoholic and alcoholic-etheral solutions unless 5 c.c. of water be present for each 0.1 gram of malic acid. If the precipitation takes place in the presence of acetic acid no malate is found with the tartrate. Sugar and other organic matter in wine hinders the precipitation of acid malate.

H. D. R.

On the (so-called) Penmetier's Method for Butter Analysis (Microscopic Examination by Polarized Light). A. Pizzi. (*Staz. Sper. Ag. Ital.*, xxiii., 38.)—The author points out that previous experimenters, including himself, had used simple

mixtures of butter and margarine (*c.f.* ANALYST XVII. 193.); he now studies various conditions which occur in practice, in order to see if they modify the results obtained.

He finds that keeping butter for some time at a temperature (28° – 29° C.) but slightly below its point of fusion, does not modify the results.

The semi-fused portions around the section made by a warm knife gave somewhat doubtful indications.

Butter exposed to the rays of the sun, and melted by this means, behaved similarly to butter fused by heat.

Various substances were added to butter; of these—

Boric Acid	}	gave	{	Suspicious indications.
Borax				" "
Cane Sugar				" "
Tartaric Acid				" "
Salicylic Acid	}	"	{	Crystals of colour different to field (with selenite plate.)
Sodium Salicylate				
" Bicarbonate				
Milk Sugar				
Glucose				
Shavings of horny substance		"	{	Grains and fragments red, some showing two or three colours.
Starch				Play of colours.
Saffron	}	"	{	The characters of genuine butter not altered.
Annatto				
Turmeric				

A quantity of milk skimmed by Laval's separator was mixed with a quantity of margarine by Jespersen's Emulsor; this was mixed with cream and separated, and was then churned into butter, which contained about 30 per cent. margarine; this mixture behaved as genuine butter.

Freshly made, unwashed butter, butter kept under water for five months, and butter prepared from clotted cream all gave results as for genuine butter.

Rancid butter covered with fungoid growths behaved as margarine; rancid butter free from fungus appeared as margarine, when the outer portion was examined, and genuine when care was taken to use the inner part.

The author concludes that the method is sufficiently exact to prove the presence of margarine mixed with butter, but it is far from being of universal application on account of the many exceptions.

H. D. R.

The Direct Determination of Nitrogen in Nitrates. A. Devada. (*Chem. Zeit.*, 1892, xvi., 1952.)—The method of reducing nitrates to ammonia by treatment with powdered metals, such as zinc and iron, in alkaline solution, is of considerable

antiquity, and gives good results if conducted sufficiently slowly. The same is true of Stutzer's method, in which aluminium foil is used, the rate of attack being slow in the dilute caustic lye that is employed. This inconvenience has been overcome by the author by the use of an alloy of aluminium, copper and zinc, which is easily powdered and has a powerful reducing action. The alloy, which can be obtained from T. Srpek, of Vienna, contains 45 per cent. of aluminium, 50 per cent. of copper, and 5 per cent. of zinc, which in dissolving leaves the copper in a finely divided state, so that it ensures regular distillation without bumping.

The process is carried out as follows: The solution, containing the nitrate in quantity equivalent to about 0.5 grams of potassium nitrate, is placed in a flask having a capacity of about a litre, diluted with 60 c.c. of water and 5 c.c. of alcohol, and 40 c.c. of caustic potash solution of sp. gr. 1.3 added. 2–2.5 grams of the alloy described above are introduced, and the flask attached to a condenser with a receiver containing standard acid. The connection between the flask and the condenser is made by means of a tube having on the limb next the flask a bulb filled with glass beads to prevent the contents of the flask splashing over into the receiver, and on the other limb another bulb to prevent the acid in the receiver finding its way into the distillation flask, should regurgitation occur. When the flask has been thus connected with the condenser it is gently heated for half-an-hour, at the end of which time the evolution of hydrogen will have slackened or ceased, and then the distillation is begun, at first cautiously, until the zinc of the alloy has completely dissolved, and then more vigorously, the time necessary being about twenty minutes from the time when the contents of the flask begin to boil. The distillate is caught in standard acid and the ammonia determined by backward titration in the ordinary way. It is to be noted that the strength of the alkali used is of importance, as if it be too strong, the action on the alloy is unduly vigorous at the beginning of the operation, and if too weak the contents of the flask have to be heated overmuch, the result in both cases being the formation of a fine spray of caustic solution, which is very difficult to stop, even with complicated attachments to the distilling flask. The test analyses on pure nitrates are satisfactory.

B. B.

Qualitative Examination of Iodine for Cyanogen. C. Meineke. (*Zeits. anorgan. Chem.*, 1892, II. 165, through *Chem. Zeit.*)—According to the German Pharmacopœia, cyanogen can be detected in iodine by rubbing 5 grams of the sample up with water, filtering and reducing the solution with sulphurous acid, adding a very small quantity of ferrous sulphate and a little caustic potash, warming the solution, and, after cooling, acidifying it. The formation of Prussian blue indicates the presence of cyanogen. Sodium thiosulphate may be substituted for the sulphurous acid if the iodine solution be first acidified. One part of iodine cyanide in 12,000 parts of water, or one part of cyanogen in 54,000 parts of water can be recognised by this test. The behaviour of

iodine cyanide in neutral solution supplies two other methods for detecting it. A few milligrams of KI are added to a neutral solution of iodine containing cyanogen, which is then decolorised with sodium thiosulphate solution of about centinormal strength, until the yellow colour has almost disappeared. Clear starch solution is added, which, if iodine cyanide be present, produces a much deeper blue colouration than if the iodine be pure. The object of the potassium iodide is to obtain the full blue colour with starch. The reappearance of the blue colour of the iodide of starch, but with a redder tinge than is normal, is also an indication of the presence of cyanogen. The other method depends on the formation of sulphate when iodine cyanide is reduced with sodium thiosulphate, which can be detected by the addition of barium chloride in the usual manner. The freedom of the thiosulphate from sulphate must be ascertained by a blank experiment with pure iodine. The reaction is about twice as sensitive as that obtained by the official method.

B. B.

[The official method, which is absolutely characteristic of cyanogen and depends on no indirect reaction, is, nevertheless, to be preferred.—B. B.]

The Determination of Ash in Mineral Lubricating Oils. A. Stepanow. (*Zap. imp. russk. tech. obszcz.*, 1892, xxvi., 927, through *Chem. Zeit.*)—The direct incineration of mineral lubricating oils is inconvenient, as they evolve much soot and leave a coke difficult to burn off. The plan adopted by the author consists in the use of a head fitted as closely as may be to the dish in which the oil is to be burnt, connected with an aspirator and a condenser, so that the vapours are drawn off and condensed as quickly as they are formed. 70 to 90 grams at a time can be treated in this way, and when one portion has been taken down to the coking point, another can be added until 200 to 300 grams have been volatilised, when the combustion of the small residual amount of fixed products can be completed over the blow-pipe or in a stream of oxygen. The head may be improvised out of a flask with the bottom cut out.

B. B.

Chemical Researches on Sicilian Cheese. Spica and De Biasi (*Staz. Sper. Ag. Ital.*, xxiii., 133.)—The authors discuss the different kinds of Italian and Sicilian cheeses. They draw attention to the fact that useful analyses are very scarce; for instance, the only analyses of "Cacio-cavallo" being those of Sartori (*THE ANALYST*, xviii., 17). They have, therefore, collected the results of 23 analyses made by themselves on typical cheeses: these include 12 Placentian and 2 Majorcan cheeses, and 9 of "cacio-cavallo."

Their methods are as follows:—*Preparation of the Sample.*—A square lump of about a kilogramme was cut out of the middle of the cheese; this was scraped for a thickness

of 2 c.m., and kept in a stoppered bottle; each time a portion was taken for analysis the top part was scraped off. *Water*.—5 grammes of cheese were mixed in a mortar with a glass pestle, and then dried to constant weight at 110° C. *Ash*.—5 grammes were burnt in a platinum capsule. When carbonised, the coal was washed with hot water to extract the soluble ash; the carbon was then burnt at a high temperature, and the solution added to the capsule and evaporated. *Sodium Chloride*.—The ash was lixiviated, and the chlorine, estimated by silver nitrate volumetrically, was calculated as NaCl. *Phosphates* were estimated by Spica's method (THE ANALYST, xvii., 116); the modification of adding sodium carbonate and sand, free from iron, to the cheese before incinerating, was adopted. *Fat*.—10 grammes of cheese were extracted with petroleum ether in a "Soxhlet"; the soluble acids are estimated by this method as fat. *Nitrogenous Matters*.—These were estimated as total nitrogen, proteid nitrogen, ammoniacal nitrogen, and amidic nitrogen. Total nitrogen was estimated by combustion with soda-lime; 2 grammes of starch being added to 4 grammes of cheese; proteid nitrogen was estimated by the combustion of the portion insoluble in hot water, with the addition of the lead acetate precipitate; for ammoniacal nitrogen, 5 grammes of cheese were washed on a filter with about a litre of cold water, the filtrate evaporated and distilled with 5 grammes of magnesia, and the ammonia titrated with N/10 sulphuric acid. The amidic nitrogen was estimated in the filtrate from the proteid nitrogen, after elimination of soluble proteids by precipitation with acetate of lead, the ammoniacal and nitric nitrogen, if any, being subtracted. Traces of nitrates were found in some samples of cheese. Acidity, as lactic acid, was estimated by washing 5 grammes of cheese till the washings were no longer acid, and titrating with N/10 KOH.

The means of the analyses were:—

			Placentian.	Cacio-cavallo.
Water	29.06	23.67
Ash	9.46	7.63
Fat	24.74	25.49
Nitrogenous matters	30.09	29.25
Proteids	23.71*	23.63
Nitrogen total	5.056	4.865
„ proteid	3.714	3.780
„ amidic	1.171	0.987
„ ammoniacal	0.091	0.097
NaCl	5.04	3.39
P ₂ O ₅	1.197	1.278
Acidity as lactic acid	1.55	1.74

Possibly a misprint for 23.21.

The most notable difference between these two types of cheese lies in the amidic nitrogen

[NOTE BY ABTRACTOR.—Kjeldahl's method is more suited to the analysis of cheese than that used by the authors. For much information that is not of an analytical nature, the original paper must be consulted.]

H. D. R.

Standardising Acidimetric and Alkalimetric Solutions. (*School of Mines Quarterly*, xiv., 62).—Parsons concludes from his experiments that the most accurate method of standardising is with potassium tetroxalate (volumetrically). Of the gravitation methods the determination (of standard HCl) by precipitation as AgCl is given the preference. The tetroxalate as usually obtained, frequently contains more or less acid (bi) oxalate. Using an excess of oxalic acid for the first crystallization and keeping the solution hot for an hour or more before cooling to crystallize out, are especially advised. No material gain or loss by exposure to ordinary conditions, or by drying over sulphuric acid was detected. Litmus was found to be the best indicator with it. The point taken is the distinct *appearance of the blue* (adding an alkaline solution from the burette). Cochineal, methyl orange and turmeric, are unsatisfactory. Acid potassium tartrate, strongly recommended by Börntrager (*Vid. Quarterly*, xiii., 175), was found to be more difficult of preparation, as it tends to retain an excess of acid. Börntrager's directions are not sufficient for obtaining a salt of the necessary purity.

W. J. S.

CORRESPONDENCE.

COUNTY ANALYST'S OFFICE, DARLINGTON,

March 10th, 1893.

THE STOCK NITROGEN PROCESS.

SIRS,

To the Editors of THE ANALYST.

This method, if one may judge from the character of the discussion which followed the reading of my last communication upon the subject, appears to have reached a stage at which, and beyond which, no further information is to be elicited by either argument or assertion. If it needs anything more to establish its claim to a front rank among accurate methods for the determination of nitrogen in organic bodies, it needs *work*.

May I say to analysts who intend to try this new process, that the confession of a skilled and experienced operator (Mr. Allen) that he "found it desirable," in working the Kjeldahl process as now known, "to make two or three determinations and take the highest result," is in singular contrast with the fact that this same method has been selected as a standard of accuracy by those who have had most to say in criticising my process.

And may I also say that I shall be most willing to do all I can, if applied to by letter, to help others over difficulties I have myself surmounted. Yours obediently,

W. F. KEATING STOCK.

SUPPLEMENT
TO
THE ANALYST.
APRIL, 1893.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

As already notified in our issue of last month, a special meeting of the Society of Public Analysts was held in the rooms of the Chemical Society, Burlington House, on February 15th, 1893. The meeting was convened to consider a series of resolutions drafted by the Council on the subject of certain proposed amendments in the laws relating to adulteration. There was a large attendance of members of the Society, and of other gentlemen interested in the working of the Food and Drugs Acts. In the unavoidable absence of the President (Sir Charles Cameron), the chair was taken by Mr. M. A. Adams. Amongst those present were Dr. Cameron, M.P., the Hon. H. A. de Tatton Egerton, M.P., Mr. Kearley, M.P., Mr. G. M. Allender, etc. Letters of regret at inability to be present were read from Sir C. Cameron, President of the Society, Sir Walter Foster, M.P., Mr. Brunner, M.P., Colonel Howard Vincent, M.P., Mr. H. J. Wilson, M.P., Sir Henry Thompson, Sir W. Pink, Sir Joseph Fayrer, and many others, including most of such provincial Public Analysts as were unable to be present.

The Chairman said that the meeting had been called under the auspices of the Society of Public Analysts, for the purpose of discussing a series of resolutions which, he presumed, were in the hands of all present. It would be noticed that these resolutions were of greater interest to the general public, and to traders liable to be affected by the operations of the Sale of Food and Drugs Acts, than they were to the Public Analyst himself in his professional capacity. He would remind those present that the subject which they were about to consider was altogether a modern institution. In point of fact, for practical purposes all that related to the Sale of Food and Drugs Acts was not more than twenty years old. It was not, therefore, a matter of surprise that in the first essay at legislation flaws should be found to exist, and in the first attempts at putting into execution a difficult measure technical difficulties should have become apparent. Between the legal and the technical difficulties, however, there was this great difference: that whereas, in the natural order of things, improvements in the technique of analysis were bound to follow as a necessary consequence with the progressive development of the analytical art, on the other hand, such legal flaws as existed in the Statute grew more and more conspicuous as time rolled on. And so it had come about that the Council of the

Society had had their attention drawn to the statutory aspects of the matter, and they had thought it would be a most desirable thing to invite representative men, whose interests were so largely involved in the matters treated of in the resolutions, to assist in their deliberations. In the name of the Society, therefore, he welcomed all those who had honoured it by their presence that evening; and he cordially invited them, in the first place, to take part in the discussion when their experience could elucidate any point; and, when the proper time came, to signify acquiescence in, or disapproval of, each resolution when put to the vote. He proposed, as suggested by the Council, to take the resolutions *seriatim*, to move them, then to ask certain gentlemen to second them, and after that to ask anyone who felt disposed to speak to do so. The first resolution was as follows :

“ That amendment of the laws relating to adulteration is urgently required.”

He considered this was a kind of preamble, which would hardly require much discussion. He would, therefore, with the permission of the meeting, unless someone wished to speak upon it, take that as agreed upon, and pass on to the second resolution, namely :

“ That the present Acts often operate unfairly on the retail traders, and that provision is necessary to ensure, in many cases of adulteration, the prosecution of the real offenders.”

Mr. Fairley, of Leeds, in seconding the resolution, said that the present Acts failed in their operation with respect to retailers :

Firstly, in the want of uniform working. Wherever the Acts were inefficiently worked, the dishonest adulterating trader could undersell the honest trader. The present system was also very unequal in its administration, as, by the vagaries of local authorities, adjacent towns or districts differed most widely in their enforcement of the Acts. In some places the food supply was well looked after, and the magistrates inflicted fines having some real relation to the magnitude of the profits derived from the offences committed. In others, the inspection was inefficient, and the present Acts neither provided nor suggested any means for securing that inspectors had some reasonable knowledge of their work, or that they should do that work. In some places the magistrates also inflicted fines which one day's fraud would be more than sufficient to cover. He thought it was worthy of consideration whether a minimum as well as a maximum fine should not be fixed by law for certain classes of offences.

Secondly, there was the incidence of responsibility. The retailers often complained that the present Acts pressed unduly upon them. A retailer who sold an adulterated article of food, whether he knew that it was adulterated or not, injured the public, and was made liable to a penalty. In common law, as anyone found in possession of stolen goods was liable to a penalty—and as in all cases of theft every effort was made to discover the original thief—so it should be with the adulterator of food. In his opinion, it was well worthy of consideration whether the adulteration of food and drugs should not be made an offence distinct from that of selling such food. It ought to be a dangerous thing to manufacture adulterated articles, and it ought also to be a dangerous thing to take any part in supplying them to the consumer.

Thirdly, with reference to securing the identity of samples. It appeared reasonable that the vendor or person supplying articles from which samples were taken for analysis should have the right to affix his own seal to the samples taken by the inspector in addition to the inspector's seal. If desired, the seal belonging to the vendor should not indicate the origin of the samples. The more perfect and efficient any acts of this kind could be made, the more the honest traders had to gain by them; and he thought that no one would contend that the present Acts were not capable of great improvement in nearly every point connected with their working.

Mr. Henry Gray could not agree with Mr. Fairley as to the fine inflicted for a day's fraud being covered by a day's profits. He (Mr. Gray) believed that Mr. Fairley could not understand very much about the profits made by retailers if he concluded that that was the case. He thought that the hardships of the retailer were caused, not by retail men themselves, but by the wholesale vendors or the packers of most of the articles. That ought to be made clear, and he hoped the point would be discussed.

The Hon. Alan de Tatton Egerton, M.P., said he would give the meeting his experience as chairman of a committee whose duty it was to see that the Sale of Food and Drugs Acts were properly carried out. In the early stages of his connection with the committee, he remembered a prosecution being instituted by the Vestry of St. George, Hanover Square, in a case of pepper adulteration, and he remembered making a calculation for his own satisfaction, by which he showed that the producer of the article—the packer, as Mr. Gray had called him—was making a profit out of the adulteration of no less a sum than £1,200 a year. He knew that in many cases it was the practice of the wholesale trader to pay the fines inflicted; he took the chance, and the law of chances, as was well known, was very much in favour of those who played the game. It was self-evident that where the wholesale trader could see his way to a gross profit of £1,200 in a year, he could very well afford to pay fines even at the rate of £5 a week, and still be a great gainer. A change in the law as recommended in the resolution was very urgently needed; and he was clearly of opinion that the reform suggested by it must be for the benefit, not only of the retailer, but, what was still more to be desired, of the general public.

Mr. Charles Estcourt, of Manchester, inquired whether this particular resolution was applicable to producers out of the kingdom. Was it suggested that the producers in foreign countries should be pursued? He stated that although he had read of this as a suggested amendment, it was evident that our Food and Drugs Act would not run out of this kingdom. He also stated that under the Margarine Act there was now no difficulty in reaching the importer or wholesale merchant. Indeed, in Manchester, a week or so before, the inspectors had visited a wholesale merchant, and had taken samples from unmarked firkins, and these proving to be margarine, a conviction ensued.

The Chairman thought that it would be impossible to prosecute the original offenders when they resided outside English jurisdiction.

Mr. Estcourt said that in Manchester, if there was an absolute warranty given, and if it proved to be a false one by the fact that the Public Analyst certified the sample to be adulterated, the giver of the warranty was always prosecuted. But

a difficulty was that warranties were often not given, and that therefore the wholesale vendors were unreachable. That state of affairs should be guarded against in any amendments of the Act, and could easily be met by following the lines of the Margarine Act, by which an invoice became itself a warranty.

Mr. Adkins did not know whether it was competent for visitors to make suggestions for altering the existing state of affairs. He had come to the meeting with the intention of listening only; but as so few suggestions had been made, he would like to make one—namely, that the retail dealer and the wholesale dealer should be made co-defendants in the case. Then, when they were both before the magistrate, it would be possible to determine whether the wholesale or the retail dealer was the real offender.

Dr. Charles Cameron, M.P., said that he had been made an honorary member of the Society of Public Analysts in consequence of the part he took in the passing of the Act of 1875. He had at the present time a Bill before the House which was intended to meet certain difficulties in the Sale of Food and Drugs Act; and, among other things, he believed it would perfectly meet the difficulty at which the resolution was aimed. He proposed that every person who sold foods or drugs should sell them with an implied warranty; that the invoice should be a warranty, even on a verbal order, unless the vendor gave a written declaration to the contrary. The meeting would be interested to hear that the Federation of Grocers was ardently advocating a change in the law. Certain of the wholesale producers, such as the Coffee-Growers' Association, were also supporting the proposal. In connection with this measure, he (Dr. Cameron) suggested that the wholesale dealer should have his goods open to inspection in precisely the same manner as the retail dealer; and he was glad to say that Mr. Colman—who might be considered as the leading mouthpiece of the wholesale men—admitted that this proposal was reasonable. It would hardly be necessary to say that Mr. Colman did not agree with some of the other proposals of the Bill; he did so on the ground that it would involve him, and wholesale dealers generally, in a large amount of trouble; but he (Dr. Cameron) believed the effect would be to make the wholesale dealers more careful. Mr. Estcourt had asked whether the foreign wholesale dealers could be got at. Of course they could not. If the first man who sold the goods in this country was made to sell them with an implied warranty, he would thus be got at, and he would not then buy an adulterated article from the foreign producer. It would pay the wholesale man to have an analysis made of the goods he bought. If a dealer ordered pepper, and a man from abroad sent him a mixture, he had his remedy in not paying, and in leaving the other to sue him. He found so much interest taken in the proposed amendment of the Sale of Food and Drugs Act, and such a wonderful agreement so far as certain general principles were concerned, that he thought it would be quite feasible to get a Bill read a second time without opposition, on the understanding that it should go before a Select Committee. If that were done, all the parties interested would have an opportunity of representing their views; new clauses could be proposed and thrashed out, and everything would be put in a fair way for the matter to be taken up next session, with every prospect of its being carried to a successful issue.

Mr. E. Hudson Kearley, M.P., claimed to have some right to speak on the question, as occupying what might be termed a hybrid connection with the sale of food products. It would, perhaps, seem rather a curious anomaly that a man could claim to be a producer, a wholesale dealer, and a retailer, but, without going into details, he would say that he claimed to be in this position. In connection with the working of the Sale of Food and Drugs Act, he wished to say first that he believed it was generally admitted that the working of the Act was intended, primarily, to protect the interests of the consumer, and the drawback was that the Act was not being so thoroughly enforced as it might be. Of course, every person who came within the operation of the Act and suffered from some short-coming felt aggrieved; the retailer complained at times that he had sold an article believing it to be pure, and that, consequently, a hardship was being inflicted on him for the misdeeds of another person, over whom he had no control. He (Mr. Kearley) admitted that much could be said in that direction; but the retailer himself in very many cases was a flagrant and guilty offender, and as the nearest source of distribution to the consumer was naturally the retailer, he thought that it was very essential that the Local Authorities—such as they were—should be called upon to enforce the Act in a thoroughly sound manner. He would give some few details of importance, which were within his own cognizance, as to how the Act was being carried out in places where, if the Local Authorities performed their functions properly, it could be better carried out, especially in regard to the sale of margarine as pure butter. The cases could be well authenticated. The law had become a dead letter by reason of the Local Authorities failing to put it in motion; a Trade Protection Society had been formed, and the following were the results of their investigations. In Wandsworth a man was found to have been selling margarine as pure butter for some months. The Association took samples, and had them analyzed, and they communicated with the Inspector of Nuisances, as they understood he was the proper person to apply to, but apparently no steps were taken.—They had a similar case at Gravesend, and were afterwards informed by the inspector that a sample had been taken at their instigation, but was found to be pure. On a further sample being taken by the Association the same week, it was found to be largely adulterated.—A well-known tradesman at Tunbridge Wells had been repeatedly summoned and heavily fined, but never by the Local Authority. On a very recent occasion the amount of the penalty was £10, the case causing a great deal of local sensation. This man immediately plastered his window all over with posters, "Pure Butter, 1s. per lb.," but continued to sell margarine as such. He was now being summoned again, but had attempted to evade his liability by stating that it was his manager's doing. This man had a branch in another town, and was there summoned by the Association. He wrote to the officer whose name appeared on the summons offering him £10 if he would withdraw the summons. The letter was produced in court, and a fine of £15 was imposed.—A member of the Association recently purchased a business in the neighbourhood of Notting Hill, and employed the late vendor's manager. The latter at once stated that everyone in the road sold margarine at 1s. per pound as pure butter, and he wished to do the same. The new proprietor, however, declined to do so, and contented himself with supplying as good a butter as he possibly could

at a cost of 110s. to 112s. per cwt. His trade fell off, and he then called the inspector's attention to the matter, but was told to let others alone, and he would be let alone himself. This shop had, owing to this unfair competition, to be closed. — At Brixton a man had for a long time been in the habit of selling margarine as butter, and repeated demands were made to induce the proper authorities to take the matter up, but without avail. The Association sent their officer, who took a sample, and divided the same, when the man rushed at him, swept the samples off the counter, and told him to do his worst, and also told him that if he came there again he would be ill-treated.—A member of the Association referred to was recently proposing to buy a shop in the Holloway Road. The would-be vendor stated that he invariably sold margarine as butter, and that he did not fear any inspector. This same member also has a shop in Upton Park, and one of his employes left him to go to an opponent who was about to open a new branch. This man told his late fellow-employes that his present master was not so opposed to his own interests as his previous one, and had no scruples whatever as to honesty, that he never sold anything but margarine as butter, even at 1s. 2d. per lb. The Association then took samples, and finding the facts of the case to be as stated, summoned the offending tradesman, who went into the box, and his wife and son also, and they all swore that it was purely an accident, whereupon the magistrate dismissed the case, with costs against the Association.—Mr. Kearley said that it had been proposed to consider all goods coming from the wholesale dealer as being sold under an implied warranty. The only objection he (the speaker) had to this was, that supposing the retailer proved to be dishonest, how would it be possible for the wholesaler to know that the article sold retail was part of the identical one supplied by him, and for which he was supposed to be responsible?

Dr. Cameron explained that as the law now stood, if a retailer produced a written warranty from the wholesale dealer he was exempt, and the prosecution must be against the wholesaler. The only difference that he made was to suggest the acceptance of the invoice as a warranty.

Mr. Kearley would suppose that it was incumbent on the retailer to obtain a written warranty in order to be exempt. How was he to know that the sample supplied to him by the wholesaler was the identical one referred to. There was another suggestion made which he (Mr. Kearley) thought most appropriate, and might tend to get rid of a great many evils; that was, that inspectors should have the most free access to the warehouses of the wholesalers. He could speak as a large wholesaler, and he would maintain that it was only fair in the interests of the community that the inspector should have just as free access there as the inspector of factories had in factories. He should be empowered to go whenever he liked to take samples from the bulk, and the mere knowledge that an inspector might call at any moment would prevent adulteration going on.

Dr. Cameron remarked that a clause to that effect was contained in his Bill.

Mr. Kearley said it had been suggested that the foreign adulterator should be hounded to his den, and the opinion had been expressed by the Chairman that that was practically impossible. It was, indeed, impossible to go to his lair and find him there in the midst of his guilt; but he (Mr. Kearley) would not admit for a moment

that there were not means whereby he could be struck at a distance, for every article that was shipped from abroad must arrive at a port of entry, where it could be subjected to the examination of the Customs officials to see whether it was contraband. He thought that at that point the Customs authorities, in association with those officially interested in the prevention of adulteration, might make periodical examinations—continuous inspections, if thought necessary—so that adulteration might be stamped out or that the penalty might be paid. That would involve no loss to the consignee, because he would be only the recipient of the goods. The man who shipped the goods would be the sufferer, because the authorities on this side would confiscate the goods. He thought that the exporter from abroad would give up his evil practices if that were enforced. He was delighted to see that there was now some public interest taken in the subject. There were other Members of Parliament who had the question very much at heart. He would suggest that the Public Analysts, who were most concerned with the carrying out of the Adulteration Acts, should assist in incorporating into any Bill which might come before Parliament such provisions as would render it almost impossible for adulteration to go on at all. Public Analysts must necessarily possess greater knowledge than Members of Parliament, and, therefore, if there was a need, as was apparent from the unanimous expression of opinion at the meeting that evening, for the evil to be scotched if not killed, he would say that, with the combined knowledge of Public Analysts, and the desire to safeguard the interests of traders generally, they would succeed in getting the Acts to work in an amended form if necessary—and consequently confer benefit on the consumer.

The Chairman then put the second resolution, which was carried unanimously.

Mr. Pattinson (Newcastle-upon-Tyne) seconded the third resolution, which read as follows :

“That in view of the fact that, as is shown in the Local Government Board reports, the Food Acts are practically dead letters in a large area of the United Kingdom, it is necessary that adequate provision be made for securing uniformity in their application and in their due enforcement.”

He said that in the interests of the public the reform suggested was very much needed. According to the report of the Local Government Board, there were 27 boroughs and 4 counties in the United Kingdom in which the Act was completely ignored, and a great number of boroughs availed themselves of it to a very limited extent. He considered that an absurdly inadequate number of samples were sent to Public Analysts for analysis. This state of affairs was not due to there not being any necessity for carrying out the Act, because even now, of the samples sent in, at least 12 per cent. were found to be adulterated. In the districts where the Act was ignored there was no doubt quite as much adulteration, and probably far more. There were several reasons why the Acts were not carried out in those places : some wished to avoid the expense ; in others the Authorities were composed mainly of tradesmen, and it was scarcely in human nature that bodies so constituted would be very active in trying to bring prosecutions upon themselves. Both in the interests of the public and of all honest traders, he thought the time had come when some

authority should have the power of insisting that the Act should be carried out thoroughly throughout the country.

The Hon. Alan de Tatton Egerton, M.P., said he would like to be permitted to move, not an amendment, but an addendum to the resolution; viz., to insert in the last line after the word "application" the word "standardizing." The object was to get at some means by which Public Analysts could know where they stood. At the present time there was a very great difficulty in carrying out the Acts with regard, for instance, to milk, as one of the most important matters, and with regard to butter, as another important matter, both as to the amount of fat in the case of milk, and as to the amount of water to be contained in the sample in the case of butter. There were other similar and, perhaps, subsidiary matters, such as pepper and mustard, and various other things which were to a certain extent mixtures; but he thought it was very important that there should be a standard fixed. At the present time—owing first to the proceedings of Somerset House, and secondly to the action of the magistrates following upon that—the standard by which milk was judged had been whittled down, until at the present moment it was next to impossible, except in very flagrant cases, to get any convictions at all. The same remarks applied equally to butter, in so far as adulteration with foreign fat was concerned, and more especially as to the amount of water it contained. There was another subject which was now cropping up, and coming very much to the front, and that was the question which was of special interest in his own county of Cheshire—he referred to what were technically known as "filled cheeses." All these subjects required the most careful consideration, and at the present time Public Analysts and the authorities appointing them were not supported by the one Authority, if the word could be used, by which they ought to be supported—the Authority set up by the Act, viz., Somerset House. Nothing short of the strong opinion of Parliament would ever bring upon the Minister concerned the pressure which ought to be brought upon him, in order that there should be a Departmental Committee, assisted by the best-known Public Analysts of the country, to establish, once and for all, what were to be fair standards by which all these prosecutions were to be conducted.

Mr. Cassal thought the introduction of the suggested word would strengthen the resolution; and while it might be advisable to discuss the matter under No. 5, there could be no serious objection to introducing the word. He seconded the amendment.

Dr. Cameron deprecated the introduction of the word. The question of standards was fought out very fully when the late Bill was before the House. It possessed the drawback that if a standard were adopted for milk, the standard of the lowest milk must be taken.

Mr. Otto Hehner: No.

Dr. Cameron said he could show that Public Analysts objected to the fixing of a standard. Lowest standards might be fixed, but it was a question which would have to be fought out. He had at one time been entirely favourable to standards, but the reasons which were adduced against the principle had caused him to entertain a hostile opinion to any attempt to standardize.

Mr. Otto Hehner said it had been asked, Why were these Acts not enforced?

Whose fault was it? Clause 10 of the Sale of Food and Drugs Act made it incumbent upon all vestries and district and county and borough authorities to appoint one or more Public Analysts. The Local Government Board had, in spite of much resistance, for years brought pressure to bear upon various authorities, and had compelled them to appoint Public Analysts, and the result was that 235 appointments had been made. But there was nothing whatever in the Act of 1875 to force the Authority to apply it. It was absolutely essential that the law be amended, and that it should not be left to Local Authorities to apply or neglect it just as they pleased. The Analyst had nothing whatever to do with the carrying out of the Acts, except to analyze the samples which were brought to him. He had nothing to do with the collection—he did not even know where the samples came from; and, in fact, he was absolutely in the hands of the Authorities above him. It was very seldom that the inspector had *carte blanche* from his Council to do as he pleased. The inspector mostly collected samples when he was ordered to do so either by the Town Council, the Food and Drugs Committee (if there was one), or by the Sanitary Committee. Not unfrequently a resolution was passed by the Council, or the Committee, that the inspector should collect a certain number of samples, thereby conveying sufficient warning to all whom it might concern. The inspector, therefore, was also powerless, although in his (Mr. Hehner's) opinion he often might do a great deal more than he did. An inspector, as a rule, was saddled with many other duties. In counties and in small boroughs it was very often the case that the police, or the inspectors of weights and measures, acted as inspectors under the Sale of Food and Drugs Act. If a tall police-sergeant went into a country village shop and asked for a pennyworth of mustard—which was a not very usual occurrence in the ordinary course of business—then, of course, the dealer was forewarned, and he took uncommonly good care to supply him with a genuine article. He (Mr. Hehner) knew from his own experience that this applied to very many other articles. It was a farce to expect that men who were well known in a rural district should succeed in putting down adulteration, however anxious they might be to carry out their office in an efficient manner. What was wanted was a system whereby proper inspectors would be appointed; but as the Acts were worked at present these, if appointed, would have nothing to do. It might appear at first sight that in many cases the Acts were not enforced because the Councils in towns and cities contained members—as they frequently did—who were directly interested in the sale of food and drugs. If, however, the reports of the Local Government Board were referred to, it would be seen that about the same proportion of County Councils, which were mostly composed of county gentlemen, failed to apply the Acts just as much as the Town and City Councils, which often largely consisted of shopkeepers. What the reason was, he acknowledged, he could not fathom. The delinquency, so far as the counties were concerned, was greater than would appear at the first blush. It could be shown that year by year the Act was being more worked. From the year 1877 to the year 1881 only 16,668 samples were taken, or one for every 1,520 of the whole population of England; in the next year (1882) 21,772 samples were taken, or one for every 1,237 of the inhabitants; from 1887 to 1891, 26,846, or one for every 1,060 inhabitants; and in 1892 29,028 samples were taken, or one for every

999 inhabitants. But, as a matter of fact, that progress was due to the spirited action of a very few Authorities, as he would proceed to show. There were a few districts which carried out the Act honourably, and they brought up the average. But, then, what about the boroughs and counties which did not apply the Acts? It appeared from the last annual report of the Local Government Board that, of 55 administrative counties in England and Wales, only 13 had worked the Act at all fairly, *i.e.*, in accordance with the requirements of the Local Government Board—requirements which were entirely theoretical; namely, that one sample should be taken for every 1,000 inhabitants. Eleven other counties had taken one sample to every 2,000 inhabitants—about half as many as, in the view of the Board, they ought to take; whilst the majority of 30 counties either did not apply the Act at all, or contented themselves with a dozen samples per annum or less, which was only just sufficient to prevent their being stigmatized in the Government reports as malefactors to the inhabitants. Thus, in Wales, with the exception of Glamorgan-shire, not one county authority had done anything that might pass for its duty to its inhabitants. In the whole of Wales, which contained a rural population of 820,000, 174 samples were taken per annum. Then, with regard to boroughs, out of 131 given in the report, only 39 had worked the Act at all honestly, another 39 had made a more or less weak attempt to the extent of about one sample to every 2,000 inhabitants, and no less than 61 had taken no action at all, or only the pretence of action; 27 had never taken a single sample. As the hon. member for Devonport was present, he thought it right to inform him that Devonport was one of those places. In fact, the greater portion of England flagrantly neglected to carry out the Acts. He would give a few instances of bad cases in 1892: Cornwall, with 299,000 inhabitants, was satisfied with taking 29 samples per annum; Monmouthshire, with 197,000, took 16; Norfolk, with a population of 288,000, took 35 samples; Northumberland, having 260,000 inhabitants, took only 28 samples; and Oxfordshire, with 127,000, was content with the small number of 14 samples; whilst Devonshire, Derbyshire, Hertfordshire, Nottinghamshire, Shropshire, Wiltshire, Yorkshire, and many others worked the Acts still less—that is, had almost neglected to work them at all. From the Census returns he came to the conclusion that, out of every 100 inhabitants in the kingdom, two were dealers in articles which came under the Sale of Food and Drugs Acts. The Census did not state how many establishments there were; but, for the purpose of comparison, he would take it that four persons on the average constituted an establishment; it would follow that for every 200 persons there was one shop vending food, drink, or drugs. The Government recommended that one sample should be taken for every 1,000 head of population; so that at this very modest figure each shop would be visited once in five years. When it was considered that milkmen changed their milk, or got new supplies twice a day; that each grocer dealt in a multitude of samples subject to the Acts which were frequently renewed, it became obvious that the requirements of the Local Government Board were so absurdly small that they did not meet the case. It could only be the sheer blindness of the Authority which prevented their doing their work. In most cases, also, magistrates looked upon adulteration as a small matter. A small dealer, say, is summoned for having sold a pennyworth of milk containing 10 per cent. of added

water; the magistrates calculated that one-tenth of a penny was not really very much of a fraud. They generally looked upon the offences in proportion to their actual money value, without touching upon or apparently even understanding the broad question of the good of the country. In the three classes of butter, milk, and spirits alone, he (Mr. Hehner) calculated, upon the very lowest computation, that the country lost every year, or paid to somebody who ought not to have it every year, about £2,000,000. The value of the imports of butter into England, so far as he could make out, amounted to something like £12,000,000 worth per annum, and of margarine about £3,000,000. What the value of the milk-supply of the country was he would be afraid to say; but anyone could calculate from these quantities, and the percentages of adulterated samples found, how immense the loss was to the country by the adulteration of these articles. If magistrates would only look upon the broad side of the question, and not at the trivial one, and would make up their minds to stop the evil, he believed they could render enormous assistance in putting a stop to adulteration. In his opinion, by far the greater bulk of the fines inflicted under the Act were ridiculously small—they were often as little as 5s. or 2s. 6d.; and when a Local Authority had for some time set the Acts in motion, and had had offenders brought up in the police-courts, and when the heavy machinery had been brought into motion and a fine of 1s. was imposed, they thought it was not worth while to work the Acts any more. These were the main reasons why the Acts largely failed in their objects.

Mr. Thomson wished to draw attention to the question of introducing the word "standard." The matter was one which required discussion.

The Chairman thought the matter could be dealt with under the fifth resolution.

Mr. E. W. Voelcker thought the matter could be made very much clearer by inserting the words "uniformity in." He thought it desirable that the word "standardizing" should not be introduced.

Mr. Cassal thought that an amendment might be inserted in resolution 5, and then the words "uniformity in" might be used.

Dr. Frank L. Teed strongly objected to standardizing at all.

Mr. Pattinson could not see any objection to the words "uniformity in" being embodied in the resolution.

The Chairman suggested that the question of "standard" had better be postponed until resolution 5 was under consideration.

The Hon. Alan de Tatton Egerton, having withdrawn his amendment, the third resolution was then agreed to.

The Chairman then called upon Dr. Turner to second the fourth resolution, which read as follows:

"That in order that the Local Government Board should have better control over the working of the Acts, a portion of the expenses of working them should be borne by the Imperial Revenue."

Dr. George Turner hoped that if he slipped out of one resolution into another the meeting would forgive him. The resolution which he had the honour to second was one of considerable importance, because he believed that upon it the whole of the benefit to be derived from any alteration in the Acts would hang. Some people

might be disposed to think that it would be better if each district were to work on its own responsibility. But there were certain things that people did not do well for themselves. If, for instance, a man required to have a tooth extracted, he generally asked someone else to put the forceps on. There was no doubt that the whole matter hinged on the question of money. Local Authorities and County Councils could not be trusted to carry out the Acts and to make the appointments under those Acts. They tried to cut down the expenses, consequently it was not a certainty that the men who were selected for these appointments were perfectly fitted for the work. Sometimes they were not. Again, Public Analysts were shamefully badly paid. It was wonderful how cheap chemistry was. If the matter were left in the hands of the Local Government Board, and it was made essential that a certain number of samples should be taken per head of the population, it would be of great benefit to the country. County Councils were not capable of seeing the benefits that lay in the Acts. The amount of money which went out of this country as payment for things which might very well be raised here by small farmers was something very considerable. He was perfectly sure that the agricultural depression in this country was largely due to the fact that the farmer did not bestir himself to pick up that which was beneath his feet. Some articles coming from abroad were scientifically adulterated, and the manufacturers there could always cut the throats of the producers in this country by adulterating down just below the price at which the article could be honestly made. There could be no doubt that scientific processes of adulteration abroad were lowering prices in this country to an extent which made it hardly worth while to produce. There was a Margarine Act, which made it compulsory that every sample of margarine should be labelled as such. He could go through twenty or thirty villages in almost any part of the country, and he would never see a sample of that commodity. The reason was very simple—it was sold as butter; and everybody who knew anything about the subject knew that margarine was disposed of in this way. It was essential for the agriculturists that they should have cheap agricultural labour, but if the labourer was to be cheated out of 25 per cent. of the profit of his labour, and if he were getting 12s. a week, and was cheated out of 3s., the retail trader was simply being paid 3s. a week out of the labourer's earnings, and the labourer got the 9s.

Mr. M. Henry said that he would be very sorry to see a portion of the expense borne by the Local Authorities. He considered that if an Act of this halting character were passed it would be just as farcical in its application as the present one. Only an absolutely compulsory Act, for which the Government bore the cost and collected the fines, would meet the case. If the fines inflicted by magistrates were contrasted, it would be seen that if a publican was summoned for selling adulterated beer, a fine of £25 might be imposed; whereas, if, on the other hand, a case of adulterating a sample of butter was taken, the fine might be 5s. or 2s. 6d. In the one case it was a Government-engineered prosecution, instituted by the Government for revenue purposes, and the best legal experts were brought to support the prosecution; in the other case solicitors who half the time did not know their business—and who were not trained to it, either—were employed. He could give an instance, within his own knowledge, where a man boasted that, in one shop in the North of England, he made a profit of £20 a week out of selling margarine as

butter ; and that man had a great many shops throughout the country, and the Local Authority where he swindled the public had never yet instituted a single prosecution. He (Mr. Henry) did not believe that the Act would be enforced in a more satisfactory manner if the Local Authorities bore half the expense of doing it. He was not speaking from the Public Analyst's point of view, but from that of the consumer. He moved as an amendment, "That this meeting asks that there shall be a compulsory application of the Adulteration Acts by Local Authorities under the direction of a Ministry of Public Health."

Mr. Angell referred to the manner in which some retailers labelled their articles. He knew of a case of an article being labelled thus : "This is sold as a mixture of whatever it shall contain." The substance referred to was lard.

Mr. Cheshire thought that the Act could be made a great deal more efficacious if Public Analysts had a little more to do with the working of the Act. He knew that, in some districts at any rate, the Public Analyst made the suggestion that so many samples should be obtained. If the Public Analyst had more liberty in that direction he could make valuable suggestions to the inspectors. He also thought that if the Local Government Board had a little more control in that way, Public Authorities would be able to get at the wholesale traders more easily. That was done to some extent at Hastings. If a retail trader complained that he was being supplied with an adulterated article, the inspector was ready to go to his shop and take a sample at the time the goods came in.

Dr. Newton thought that, if Public Analysts had the courage of their opinions, they would insist that the inspectors should be under the control of the Public Analyst. The taking of samples was half the battle, and if a man who was known to the shopkeepers of a town was employed, there would be no necessity for a Public Analyst, because the shopkeepers would always give an inspector who was known a genuine sample.

Mr. Henry said that the resolution suggested that the Local Government Board should be asked to contribute a portion of the expense. He thought that the Local Authorities would not be very prompt in coming forward with the other portion. His amendment was that the working of the Act should be made compulsory.

Mr. Cassal said that the point was not clear. Did Mr. Henry suggest that a portion of the expenses should be paid by the Local Authorities, and a portion by the Local Government Board? or did he suggest that the whole should be paid by the Local Government Board, or the whole by the Local Authorities? As a matter of fact, under the Acts Public Analysts were really officers of the Local Government Board, as well as officers of the Local Authorities.

Mr. Henry replied that he suggested inspectors should be appointed, and that they should be sent round the country just as the Excise inspectors were.

The resolution as printed was then put to the meeting, and carried.

The fifth resolution was as follows :

"That in view of this, it is desirable that there should be a duly constituted Chemical Department of the Local Government Board, with whom the Public Analysts, as officers of the Local Government Board, should be placed in direct relation."

Mr. Alfred H. Allen, in seconding the resolution, said that counsel were of opinion that there should be an official Chemical Department, which did not exist at present. It was proposed that there should be a Chemical Officer attached to the Local Government Board. There was already a Medical Department, which gave authoritative decisions on public medical questions, but the chemical side was left unrepresented. He constantly received from the Canadian and American Governments most valuable blue-books upon various matters connected with the Food and Drugs Acts of those countries, but the Department here did not take the trouble to send out such books, with two exceptions, to their own officers. He only knew of the reports issued by the Local Government Board by hearsay. Although his own reports, and those of other British Public Analysts, were quoted in the returns of the Local Government Board, he and they were not in touch with the Department. It was a fact that legislation was projected and was actually being carried out in America to stop the adulteration of lard with cotton-seed-oil before Public Analysts in this country were even aware of the adulteration. He had also heard it stated that the adulteration of pepper with ground olive-stones—known as *poivrette*—was well known to a Government Department in this country; but it was not communicated to Public Analysts, who were left to find it out for themselves. If there were a Chemical Official attached to the Local Government Board, it would be his business to be in touch with foreign officials occupying similar positions; and it would be his duty, if he had reason to believe that a new form of adulteration was being carried out, and had been noticed in certain districts, to warn Public Analysts to be prepared for it. It would be found that such an official would have plenty to do to look after these and kindred matters, together with the water supply, which would be a most important duty of the Department. He did not suggest that the proposed Chemical Officer should act as a referee under the Sale of Food and Drugs Act. He thought that, if the certificate of a Public Analyst upon any sample was disputed, the Chemical Officer should be applied to, and he should send the disputed sample to an Analyst who had special experience in the particular kind of analysis upon which the certificate was based. At the time the present Act was passed the only court of reference thought to be available was the Revenue Laboratory of Somerset House, and the officials there had thrust upon them a disagreeable additional duty outside and beyond their ordinary work as Excise chemists. They had a score or two of disputed samples submitted to them annually, quite apart from their ordinary duties, and they were expected to make themselves conversant with the best methods of food and drug analysis. On that account they were placed in a false position, and one it was not fair to place them in. They had been called on to undertake duties with which they were not familiar, and had evaded the responsibility attaching to their office. An incompetent dairymaid in Ireland prepared butter containing 19 to 20 per cent. of water, and as the butter was not made for sale, the referees regarded such butter as genuine, and fixed their limit accordingly, thus making the said dairymaid the referee under the Act. He, Mr. Allen, believed that to fix standards by Act of Parliament would be a very great mistake. It was true that standards had been established in the case of spirits, but, then, spirits were capable of being analyzed with very great accuracy, and their strength could be adjusted with precision; hence they were not liable to the

variations which were common to butter and milk. Any standards resolved on should be fixed by the proposed Department of the Local Government Board in consultation with experts, and they ought to be capable of revision at any time by that Department as a central Authority. Standards under such circumstances might be tolerated; but he thought that to lay down standards for food generally would tend very largely to prevent improved processes of analysis being devised. The standards should be laid down by the best authority. The incompetent or careless dairymaid was at present the authority with regard to butter. She was the referee—and that was a thing which was not to be tolerated in future. With reference to the extent to which adulteration was being carried on, he had made a calculation, based on actual analytical results, showing that in Sheffield last year the public lost upwards of £5,000 by paying for water instead of milk.

Mr. Thomson said he had been instructed that if he certified that a certain sample of milk had been adulterated with a given quantity of water, a prosecution could be instituted; but that if he gave the precise facts and stated that the milk was adulterated with a certain quantity of water, as judged by a standard, no action could be brought against the person who sold the adulterated milk. He thought that standards should be recognised by the Adulteration Act as a means of simplifying the work of Public Analysts; then the prosecution of a person who sold milk under the standard would not necessarily prove him dishonest. He believed under the present arrangement honest men had been charged with and fined for “fraudulently adulterating their milk with water.”

The resolution was then carried.

The Chairman then called on Mr. Cassal to second the sixth resolution as under:

“That the present system of reference in the case of disputed analyses is unsatisfactory, and ought to be entirely remodelled.”

Mr. Cassal believed that the resolution which had been placed in his hands by his colleagues was, perhaps, the most difficult and delicate of all the resolutions on the paper. He was, however, considerably relieved by the fact that Mr. Allen had already dealt with the subject, and had, indeed, said enough to warrant the passing of the resolution by the meeting. That the present system was unsatisfactory, he thought most persons who had any experience of the working of the Acts would agree. The system had never worked well, and it was obvious to Public Analysts, to Local Authorities, to inspectors, and to magistrates that that system was objectionable from every point of view to all who had anything to do with the Acts, either as prosecutors, or for prosecutors, or as defendants, or for defendants. It was necessary, in order to understand the resolution, to explain the origin of the reference of samples in disputed cases to Somerset House. It had already been shown that evening that at the time of the passing of the Adulteration Act Somerset House was the only place of bottles and tubes; it was the only Government Department that was supposed to be in any sense scientific. All the others were, apparently, not scientific in any sense whatever. But the Somerset House Chemical Department, he believed he was right in saying, had originated in a Department which was engaged in the determination of the amount of alcohol in spirits and ordinary

alcoholic liquids, and in the determination of the amount of moisture in tobacco; and he hoped he would not be considered as doing an injustice to gentlemen whom he regarded personally as friends by stating what he believed to be true—namely, that for many years their scientific labours were confined to the estimations referred to. It was, however, a fact that since the passing of the Sale of Food and Drugs Act, and the thrusting of the reference in disputed cases on to the shoulders of the officials at Somerset House, those officials had laboured hard to master the processes of analysis which had been mainly discovered by the Public Analysts of the country, and which had been principally brought out at the meetings of this Society. He was prepared also to acknowledge that the Somerset House officials had made some investigations of their own which it might have been better if they had not made, and that they had stated some results which, however, they had never subjected to the test of scientific criticism at the Society of Public Analysts or elsewhere. He might, however, congratulate the officials of Somerset House on the measure of success which had attended their efforts, while he could only regret that that success had not been greater. It was an unfortunate fact that the relations existing between the Somerset House officials and the Public Analysts, in consequence of disagreements with respect to certificates, had not been cordial, as the Past President had said, ever since the passing of the Act. He would take the opportunity of correcting the assumption which had recently been made by a writer in a paper which he presumed was held by some to be an important print—namely, the *Pharmaceutical Journal*—that there was any bitter or unpleasant personal feeling on the part of Public Analysts towards the gentlemen who filled official positions at Somerset House. He very much feared that some of those gentlemen themselves thought that there was; but he thoroughly believed that all Public Analysts were most anxious to remove any impression of that kind, and that it had no real foundation. He believed that they entertained—and he on his part most certainly did entertain—the greatest personal respect for Dr. Bell and for his colleagues. He (Mr. Cassal) considered that their views and proceedings, while often mistaken, were quite conscientious. Having said this, he must state his belief that all Public Analysts were strongly of opinion that the Somerset House officials should have nothing whatever to do with the Sale of Food and Drugs Acts. It was no doubt difficult for his colleagues and himself to make good their position in the eyes of others who were not as intimately acquainted with the real circumstances of the case as they were themselves. It was difficult for them to fully explain even to reasonable and fair-minded men how it was that they had long held the opinion that the system of reference should be remodelled altogether, and why it was that they thought, as shown by the resolution immediately preceding, that for the Somerset House reference should be substituted a reference to a Chemical Department of the Local Government Board, to which should be attached a properly-qualified Chemical Officer, who should of necessity have been a Public Analyst of extensive experience, and who would seek, in special cases of reference, for the assistance of persons specially qualified to deal with such cases. He would point to the excellent address delivered by the Past President (Mr. Hehner) as containing a very clear statement of the case, and he (Mr. Cassal) did not need to repeat the statement. Mr. Hehner had fully proved

in his paper how impossible it was for the Public Analysts and the Authorities concerned with the application of the Acts to hope that there would ever be a change for the better at Somerset House. Somerset House had, in point of fact, "the defects of its qualities," if qualities was the right word to use. The Somerset House officials had been guilty of the much-maligned standard-fixing, and that in a manner which was quite unscientific; and, most unfortunately, they had committed themselves to particular positions on several questions, and would have to stand or fall with them. Whether in science or in pseudo-science, when a person had once committed himself to an erroneous theory or to an incorrect method, it was exceedingly difficult to make him see that he was wrong, and still more difficult to make him admit it. And it was also extremely difficult, if not impossible, to get any Government Department to admit itself to be in the wrong, upon any point whatever, even if the demonstration of the fact amounted to a *reductio ad absurdum*. It could not do so. It was not in the character of the organism; inasmuch as it had been proved up to the hilt, in the papers which had been read before the Society and elsewhere, that the course adopted at Somerset House upon certain matters was altogether wrong, and, indeed, scientifically absurd; and as, outside Somerset House, no scientific man whatever with any knowledge and experience of these matters thought for a moment that the position adopted by that Department was other than entirely untenable. The result was simply this: that on the one side there were the Public Analysts protesting against the reference of samples, upon portions of which they had presumably reported, to officials who held views which were not scientific—which were, in fact, entirely erroneous; and, on the other side, there was a Government Department restricted and confined by views which were like the laws of the Medes and Persians, and occupying a position from which they could neither draw back nor move forward. The best way out of the difficulty was the one suggested in the fifth resolution—namely, to relieve the officials at Somerset House from all connection with the Acts, and to place the matter of reference in the hands of a new authority thoroughly acquainted with the most recent scientific facts and methods, and in a position to obtain the latest information. Public authorities and Public Analysts had every right to expect these qualifications in a court of reference. He felt it incumbent upon him to state that additional difficulties had been created by a particularly unfortunate attitude on the part of the Somerset House officials. This attitude, which had shown itself on various occasions, appeared to consist in this: that the Somerset House official laboured under the impression that, as a Government official, he was in every sense a very superior person; and so much had this feeling appeared to pervade the atmosphere of Somerset House, that they had actually been told in public—certainly under the mellowing influence of the festive board—that, as Public Analysts, they had very much improved of late years; that they had gradually, in process of time, and under the patriarchal education of Somerset House, advanced to such a position that they were gradually beginning to see the error of their ways. No doubt many felt some joy on hearing this; for it might lead some persons to hope that under this protecting *agis* poor benighted Public Analysts might ultimately be able to make as wonderful analyses, and to draw from such analyses conclusions as startling, as those of Somerset House. He

believed he was right in stating that the number of samples officially analyzed at Somerset House under the Sale of Food and Drugs Act did not exceed about 40 per annum. And although he believed that recently analyses of foods and drugs had been conducted by the Department for other Government offices, to say that the officials concerned possessed a tithe of the experience which would entitle them to adopt a paternal and patronizing attitude towards the general body of Public Analysts of the country had only to be stated to be refuted as ridiculous. It was, as he thought he had shown, quite hopeless to expect any alteration for the better at Somerset House, however willing the present officials might be to effect such an alteration, and however ready any future officials might be to effect it, bound down as they were, and would be, by the benumbing shackles of red tape, and the deadweight peculiar to an old-established and somnolent Government Department. One way out of the difficulty had been shown; and without entering further into the explanations (many of which would be of a very delicate nature) that could be entered into to prove that it was highly undesirable for the officials of Somerset House to continue in their present position, and for the system of reference to remain unaltered, he trusted that he had said enough, and that he had said it without being in any way offensive, to show the importance of providing the Somerset House officials with a golden bridge over which they might pass quietly and gently, and whereby they might gracefully and completely sever their connection with the Adulteration Acts.

The resolution was carried unanimously.

The seventh resolution was as follows :

“That the compulsory combinations of the two offices of Medical Officer and Public Analyst are in the public interest undesirable.”

Dr. Alfred Ashby (Reading) said that he did not know why he had been asked to second the resolution, unless it was that he was one of the sinners who held the combined appointments of Medical Officer of Health and Public Analyst. In the first place he would say that he understood the resolution to imply, not absolutely that the two appointments should not be held in exceptional cases by one and the same person—for, if that were so, the profession might be the poorer by the loss of such men as Dr. Adams and one or two others, who had largely contributed to analytical work—but that the holding of the two appointments by one person was, as a rule, undesirable, and was necessarily becoming more so every day. The training of the medical man and the chemist were so widely different that it by no means followed that because a man was fit for the post of Medical Officer of Health he was also fit to hold the office of Public Analyst. Indeed, quite the contrary was the case. The smattering of analytical knowledge required to obtain a diploma in Public Health in no way qualified the holder as a Public Analyst. It was often thought by Local Authorities that the holder of such a diploma was fit to be a full-fledged Public Analyst. Nothing could be more absurd. It was lamentable to think how low the Public Analyst had fallen in the estimation of some Local Authorities. He had heard of a case of the Medical Officer of a large borough being offered the appointment of Public Analyst for £5 a year, and this was,

indeed, bringing the office of Public Analyst into contempt. With such appointments, the very large body of honourable traders, who were to be found in all parts of the country, must necessarily suffer, for the thoroughly efficient Public Analyst was their greatest friend. He was of opinion that no local authority should make it compulsory for the appointments to be held by the same man—no one should be placed in the false position of being Public Analyst if he was not fitted for the office: but if a Medical Officer of Health happened to be an efficient chemist, the fact of his holding that appointment should not debar him from holding the office of Public Analyst.

Dr. Turner said that he also had been Public Analyst and Medical Officer of Health at one and the same time. He was therefore very well able to express an opinion, and he unhesitatingly condemned the practice of combining the appointments. He did not think Medical Officers need object in the slightest to the office of Public Analyst being taken from them. They would find in their own special department a number of difficult problems sufficiently large to last them their lives, and they would probably leave legacies in problems, if in no other way, to their children.

Dr. Hake thought many others would agree with him that, under ordinary circumstances, the medical student had not the opportunity of obtaining such knowledge as would fit him to undertake the duties of a Public Analyst; and he thought, further, that everyone present would agree that certain gentlemen who had held the dual position were gentlemen of very exceptional attainments. Some medical men, as was well known, had been brilliant exceptions, as, for example, the late Dr. Tidy, who was an admirable chemist.

The resolution was then put to the meeting, and carried unanimously.

The last resolution read:

“That provision should be made to ensure better than heretofore the proper qualification of officers under the Act.”

Mr. Hehner seconded the resolution, and said that it was closely connected with the one just passed. When the Sale of Food and Drugs Act was passed there was no large body of men, such as the Act created, fully qualified to undertake the duties required under the Act, and everybody who had even a slight claim to the knowledge, experience, and skill which the Act required was considered fitted for an appointment. The consequence was that, in the first few years, there was a great deal of friction and more or less incompetence, with the result that the work and the value of Public Analysts had been belittled for many years; but this had now, to a great extent, died out. Since that time the science of detecting adulteration had made immense progress; nevertheless, it was infinitely more difficult nowadays, in spite of all the labours which Public Analysts had bestowed on the subject, to come to a definite conclusion as to whether a sample was adulterated than it was seventeen years ago. Adulteration had now become a fine art. Analysts were also at that time very confident that they could analyze all the articles submitted to them. In fact, prior to the passing of the Food Act, a Parliamentary Committee inquired into the means which were available to put an Adulteration Act into operation. Chemists gave

evidence before the Committee to the effect that there was no scientific difficulty whatever. If analysts were asked now if there were any difficulties they would say that there were, and great ones. At that time it rested with the Local Authority to say whether a man had the requisite knowledge, and this was still the case. In fact, the vestries and councils were actually made the judges of the capabilities of a man, though they had not the least idea what was required. Every man who had studied chemistry even in a hospital was taken to be, as a matter of course, a fit and proper person to undertake the duties required by the Act. But, in reality, to become an efficient Public Analyst, he must have, not only a certain amount of knowledge, but wide experience, which could only be gained by years of work in a proper laboratory. It was thought by the Council of the Society of Public Analysts that the time had long ago come when the councils and vestries should no longer have the responsibility of appointing Public Analysts without having reliable evidence, which they could understand, before them. At present Local Authorities had a number of applications submitted to them, and the only means of guidance afforded was by the very objectionable means of testimonials. He had seen testimonials given by persons who could not possibly be judges of the needful qualifications, from which no opinion whatever could be formed of the qualifications of a man. The consequence was that, in some districts—and especially in those where the Act was not worked—men had been appointed who were not fit for the post, and who certainly did not add to the dignity of the profession. It was essential that the electing body should know that the man they elected was as well qualified as the Medical Officer of Health, and other professional officers employed by it. Therefore he was strongly of opinion that the next Act should embody a clause to ensure that a Public Analyst was as qualified for his work as a Medical Officer of Health was for his. How that was to be done he would not enter into. Two Chemical Associations in this country were moving to ensure the proper qualification of their members. The Institute of Chemistry was doing so from the point of view of general qualification, and he might say that the Council of the Society of Public Analysts, as the only body of professional analytical chemists in England, had had under consideration, and had adopted in principle, a scheme for instituting examinations with a view to secure the efficacy of candidates aspiring to the office of Public Analyst especially.

The resolution was then put to the meeting and carried unanimously.

The Chairman said that the Society of Public Analysts, although numerically small, was highly representative, as it practically included all the Public Analysts of England; and feeling, as the Council did, that the subjects set down for discussion were of very high importance—more to the outside public and the traders than to themselves professionally—they deemed it desirable that strangers should be invited—Members of Parliament, and those who dealt in food and drugs as a business, in order that they might have the benefit of their assistance in the deliberations. He had already welcomed them in the name of the Society, and now he thanked them for their presence and the part they had taken in the discussion.

A vote of thanks to the Chairman, moved by Dr. C. R. Alder Wright, and seconded by the Hon. Alan de Tatton Egerton, M.P., concluded the business of the meeting.

THE ANALYST.

MAY, 1893.

ANNOUNCEMENT.

A World's Congress of Chemists will be held at Chicago, U.S.A., during the latter part of August, 1893. The exact date is not yet fixed. The entire field of chemistry will be covered, ten sections having been constituted. The department of *Sanitary Chemistry* has been assigned to Dr. Henry Leffmann, 715, Walnut Street, Philadelphia, for organization, and he will be pleased to receive notice of any papers intended to be read in that department, or to furnish information to those intending to contribute to the work of the section.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on the 5th April, at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Alfred H. Allen occupied the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed:—As Members, Mr. William James Orsman, F.C.S., F.I.C., Public Analyst, Wigan; Mr. Charles E. Sohn, F.I.C., F.C.S., Anerley. As Associates, Mr. A. H. M. Muter, assistant to Dr. Muter; Mr. C. Hubert Pope, assistant to Mr. Horace Sweete.

The following gentlemen were duly elected:—As Member of the Society, Mr. W. C. Williams, B.Sc., Liverpool. As Associate, Mr. H. I. Foster, Hull.

The following paper was then read by its author:—

THE LISTER-BABCOCK MILK TESTER; WITH SOME SUGGESTIONS FOR EXTENDING ITS USE.

By G. EMBREY.

AFTER the very large number of papers which have at different times been brought before the notice of this Society relating to milk analysis, it would seem that the subject had become exhausted; and the splendid process of Dr. Adams is so near perfection, that it would appear nothing is left to improve, yet analysts rightly attach much importance to simple, yet fairly accurate, methods as a means of sorting samples. The most promising of these would appear to be those depending on the solution of the curd, and setting free the fat in a convenient form for measurement. Any method which is entirely volumetric and dispenses with weighing will always command attention.

Dr. Paul Vieth (in *ANALYST*, vol. xiv., page 86) has given a good summary of the various methods of milk analysis; I will therefore confine myself to those in which centrifugal motion is employed.

The first, and probably one of the best, is the Lactocrite, the merits of which have been brought before the members by my friend, Mr. Faber, and there is no doubt it is a very reliable and accurate instrument.

One objection, and a fatal one, to its use in a dairy, or even in a laboratory in which a balance is kept, is the need of heating with a volatile acid and the enormous speed required—6,000 revolutions per minute.

In reference to the accuracy of this instrument, Dr. Vieth remarks:—"It is generally admitted that results thus obtained agree very closely indeed with those arrived at by determining the fat gravimetrically. At the same time, it is rather curious that some experimenters—*e.g.*, W. Blyth—compare it with Adams' method, while others, among them Soxhlet, compare it with the latter's method, both parties find agreeing results, and still paper and plaster methods do not give results agreeing among themselves."

I am not suggesting, nor I am sure did Dr. Vieth, that either of these gentlemen were wrong or had suited their figures to their desire, for I think this discrepancy may be explained easily. In processes of this kind different methods of working will produce different results, as I shall show, and in determining the value of a new method it is needful to have a standard; we then work our new process until the results reach our standard. For myself I take Dr. Adams' paper coil method, and alter the method of working in a new process till I reach my ideal. I think it will be generally, if not universally, admitted that Dr. Adams has given us the best method of determining fat in milk.

The other objection to the use of the Lactocrite is cost.

The next process I would refer to is that of Leffmann and Beam, whose apparatus is less costly than the Lactocrite, still, it is twice as much as the Lister-Babcock. Another serious objection to their apparatus is that the substance measured is not fat, but fat and fusel oil. It is also objectionable in consequence of the acid fumes.

In vol. xvii., page 127, *THE ANALYST*, will be found a paper on the Babcock process, by Mr. Stokes, in which the method of using the apparatus is fairly well described, but from his description of the condition of fat and the poor results obtained, there is no doubt that he adopted altogether erroneous methods, for which he is in no way responsible, as he followed the instructions sent with machine.

The instructions generally sent out with the apparatus (which are copied from the American pamphlet) direct that sulphuric acid of sp. gr. 1.843 should be used. This is the gravity in the British Pharmacopœa for acid containing 98 per cent. of the true acid, but if one asks a Pharmaceutical Chemist for a sample of pure sulphuric acid, it will have a specific gravity of 1.840 or 1.841, which, according to Otto's table, contains 98 per cent. I have no doubt that this is the reason why Mr. Stokes obtained such poor results, the success of the operation depending almost entirely on employing acid of proper strength. If too strong, the curd is charred and it is impossible to read the fat correctly; if too weak, the curd will not all be dissolved. After a considerable number of experiments, I have fixed as limits sp. gr. 1.831 to 1.834. Acid of this strength will be produced by taking nine parts of B. P. acid and one part of water. So important is this strength of acid that I have had constructed a hydrometer, with two lines marking the gravity, so that in using it it is only necessary to dilute the strong acid till the liquid surface lies between them when the mixture is cooled to 60° F.

Corrected instructions for using the Babcock tester.

Measure the milk by pipette into the bottle, holding the bottle in an inclined position. Measure out the quantity of acid (17.5 c.c.) in the measuring glass, and pour into test bottle. In pouring in the acid the bottle must be rotated in order to wash all the milk down from its neck, otherwise a clot of undissolved curd may form in the fat. Well mix the acid and milk and whirl in machine for ten minutes, giving about 60 turns of the handle per minute. Fill the bottles up to the 7 mark with water which has just ceased boiling; put some of this same water into the tank of machine: again whirl the bottles for two minutes, and measure the fat.

The first form of apparatus was well described by Mr. Stokes, *THE ANALYST*, vol. xvii., p. 127. The most serious fault in the original was the oscillation when full speed was obtained. It was certainly a clumsy piece of apparatus. Messrs. Lister have, I think, succeeded in producing a much better machine and this I have the pleasure of showing. It will be seen that it is compact and entirely overcomes the difficulties of the original form.

In examining the results and comparing with coil extraction and calculations from *Hehner and Richmond's tables*, we get what appears a serious discrepancy.

Fat by Babcock.	Babcock + '3.	Fat by Adams.	Babcock + '4.	Calculated Fat.
2.5	2.8	2.83	2.9	2.88
3.1	3.4	3.39	3.5	3.47
3.1	3.4	3.42	3.5	3.48
3.3	3.6	3.62	3.7	3.66
2.9	3.2	3.24	3.3	3.31
3.0	3.3	3.3	3.4	3.36
3.2	3.5	3.48	3.6	3.57
1.4	1.7	1.67	1.8	1.75
4.1	4.4	4.42	4.5	4.5
2.7	3.0	3.05	3.1	3.09

It will be seen that we obtain a result .3 per cent. below Adams' coil and .4 per cent. below calculated fat, and my experience has always been that fats calculated according to these tables are generally higher than those obtained by coil extraction. This was referred to by Mr. Stokes, *THE ANALYST*, vol. xiv., p. 30.

No. 1	...	2.91 Adams	3.14 Calculation.
„ 2	...	2.71 „	3.11 „
„ 3	...	2.48 „	2.95 „

The question now arises, why this difference? Mr. Stokes suggests fat particles retained by white sediment in bottle or locked up in black masses of charred curd.

I separated from several bottles the white sediment, and examined with microscope, but failed to find fat globules, the mass consisting of thin plates of selenite, which show good colours when placed between Nicol prisms. As regards the charred particles referred to by Mr. Stokes, these need not exist if the apparatus be properly worked.

How then can we account for the deficiency? This is simply due to inaccuracy in graduating the tubes; of course, like all constant errors, it is of no consequence and can easily be allowed for.

The advantages I claim for the Babcock over its predecessors are:—

1. No volatile acid required.
2. No artificial heating.
3. Pure fat alone measured.
4. Cheapness.
5. Small number of revolutions per minute required as compared with the Lactocrite (600 against 6,000).

* 6. Fat may be measured many days after an experiment by warming and whirling again.

It occurred to me some time since to use the tables of Messrs. Hühner and Richmond for calculating the total solids, but I find these gentlemen had forestalled me. I have, however, made a correction on the lines indicated, viz., reducing by .1 per cent., and carrying the results only to one place of decimals, and in this way furnished agriculturists with a simple method of determining the value of milk for cheese-making. This with the special table of Dr. Babcock for ascertaining the amount of butter available, gives all that is required by the dairy farmer, and also as a sorting method for the analyst. The limit of error I fix at .1 per cent.

I do not suggest that in the case of adulterated samples it should be used, except as a check; the elegant method of Dr. Adams should in these cases be employed.

I give the following table as showing how the results work out in practice:—

	Samples A.					B.	C.
Total solids	12.24	12.55
Specific gravity	1032.88	1032.8
Fat by Adams	3.28	3.45
„ calculation	3.34	3.61
„ Babcock	2.9	3.2
Solids-not-fat from Adams	8.96	9.35
„ „ „ Babcock (.4 added to fat)	8.94	8.95

Sample B. was obtained by taking 150 c.c. of A. and adding 50 c.c. of water. The figures obtained bear this out.

Example of solids calculated from Babcock test:—

Sp. gr.	Fat by Babcock.	+ .4	Solids from Babcock.	Solids estimated.
1024.9	3.0	3.4	10.3	10.25
1033.68	1.8	2.2	11.06	10.97
1024.6	3.0	3.4	10.16	10.02

Description of Apparatus.

Fig. 1. Original Babcock Machine. In this machine the bottles AA are fitted in tin tubes on periphery of rotating wheel. The speed is obtained by pressure of large wheel B on india-rubber pulley at C.

The chief faults are:—

1st. Centre of gravity of rotating part is too high, causing great oscillation at high speed.

2nd. The india-rubber pulley soon becomes softened.

Fig. 2. Lister-Babcock machine. This has the following advantages:—compactness, no perceptible oscillation at whatever speed it may be used; the bottles are fitted in wire cages so that a better position is secured while whirling, and when at rest they become vertical.

The speed is obtained by a toothed pin A, working inside larger toothed wheel B, the direction of motion being changed by a pair of bevel wheels C. The bottles E E hang in steel wire cages resting in slots in cast iron flange D.

Fig. 1.

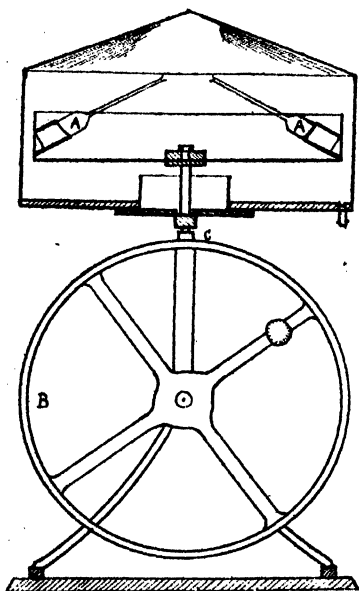


Fig 3



Fig 4



Fig 2.

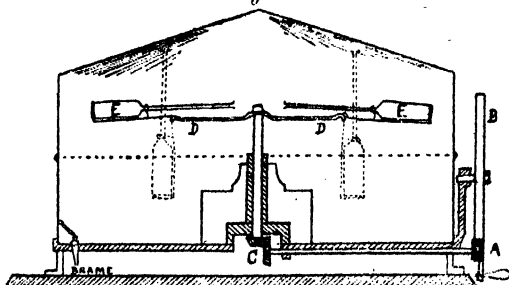


Fig. 3. One of the test bottles.

Fig. 4. Special hydrometer with one mark at 1.831 and another at 1.834 for determining strength of acid.

I must express my indebtedness to Mr. J. S. S. Brame for the drawings which accompany this paper, and also for much help in working out the most suitable conditions for obtaining reliable results. I have also to thank Messrs. Lister & Co. for the loan of apparatus and ready help on all occasions.

Since the above paper was written I have had an opportunity, by the courtesy of Mr. Droop Richmond, of more carefully examining the Leffman-Beam apparatus, and I certainly think if this be used some steps should be taken to protect the persons using it. If the pins holding bottle sockets get broken while in use (a very likely thing) the enormous speed would be almost certain to drive the liberated cup through the operator's body.

I have also learned that the bottles used in the Babcock process are calibrated, so as to give results enabling agriculturists to determine the amount of fat available for butter-making and not the actual amount in the milk.

DISCUSSION.

Mr. Allen said that the apparatus was undoubtedly an interesting one, and one which all Analysts would be anxious to experiment with. Mr. Embrey seemed to have overcome several important difficulties. He (Mr. Allen) had been prevented from using the Leffmann-Beam centrifugal method, because he had felt that he did not know exactly what he had been measuring. To be able to get the pure fat out was a distinct advantage, and it would seem that a practicable method had been brought forward. He was glad to think that Mr. Embrey had found a satisfactory explanation of the discrepancies between this method and the Adams' coil process.

Mr. Embrey stated that the apparatus had been patented by Messrs. Lister & Co., Limited, of Dursley, England, and that the price of an apparatus fitted with eight tubes was £2 10s.

Mr. Allen pointed out that Thörner, who had done much work with this apparatus, had adapted centrifugal force to several other analytical processes, employed 6,000 revolutions per minute. He had been thinking of using the method for obtaining the unsaponifiable matters from oil. Mr. Embrey had laid great stress upon the strength of the acid in milk analysis, but it seemed to him that the difference between 1·843 and 1·840 was very small. He understood Mr. Embrey to say that the acid he used was made by mixing nine parts of B.P. acid with one part of water.

Mr. Embrey pointed out that when sulphuric acid and water were used, contraction took place. He had determined the exact strength of the acid by precipitation after diluting.

Mr. H. Droop Richmond said that the density of strong sulphuric acid (*e.g.*, 99 per cent.) was less than the density of sulphuric acid when it was diluted with a small quantity of water, the maximum density being reached at 97·5 per cent.*

Sulphuric acid was in this respect analogous to the well-known case of acetic acid, but the change in the density curve was less marked.

He had examined the machine before the meeting, and it seemed a good and ingenious one. He was in the habit of using the Leffmann-Beam method. With reference to Mr.

* The following figures, taken from Mr. Richmond's tables of the specific gravity of sulphuric acid (*Jour. Soc. Chem. Ind.* ix., 1890, No. 5), show the change in density:—

Sp. Gr.	Strength.	Sp. Gr.	Strength.
1·840 ...	99·61	1·843 ...	96·93
1·841 ...	99·20	1·841 ...	95·40
1·842 ...	98·89	1·840 ...	94·96
1·843 ...	98·08	1·834 ...	92·38
1·8432 (Max.) ...	97·50	1·831 ...	92·22

Embrey's statement that it had been proved by Mr. Hehner that a mixture of fat and fusel oil was measured in the latter method, he was conversant with Mr. Hehner's paper, and it only contained a suggestion in that direction. It was he (Mr. Richmond) who had brought forward evidence in proof, but further investigation showed that this view was erroneous. He had, therefore, withdrawn it, and had entered into detail in his second paper on the Leffmann-Beam process. He was of opinion that the Leffmann-Beam process possessed advantages over the Lister-Babcock process, since instead of turning for ten minutes and stopping to pour in hot water, it was only necessary to turn for one minute. As for the acid fumes flying about, he had not observed or been incommoded by them. He had himself obtained a couple of results on a two-bottle machine under two minutes. It was very apparent that with the other process it would take a quarter of an hour to obtain a result. He had not found that the bottles of the Leffmann-Beam machine broke, even when filled quite to the top; in fact, he always filled his bottles within a quarter of an inch of the top, and always obtained satisfactory results. With the Leffmann-Beam machine it was possible to read to rather less than 0.1 per cent. He always read to a quarter of a division, which equalled 0.025 per cent. It might be said that it was a refinement to do that, but the delicacy was increased. He had published the figures of many experiments showing the great concordance. One other advantage of the Leffmann-Beam method was that there was no necessity to trouble about the strength of the acid; it could be taken as it came and used straight away. The variation in the strength of the acid made very little difference. For some time he had been investigating the estimation of total solids, and he had now come to the conclusion that the determination of the total solids was one of the least accurate estimations in milk analysis. He had found out that according to the way in which the experiment was carried out, the results could be easily varied within a quarter of a per cent., *practical* constancy being always obtained. He had made a considerable number of experiments with the asbestos method of total solids estimation, and had found that the calculated result was usually about 0.1 higher than the fat as estimated. He believed the reason for this was because in the asbestos method the total solids obtained were higher. The average total solids by the S.P.A. method were a trifle too low, and as the formula was calculated for this method, it would naturally not agree with a method yielding different results. Mr. Embrey had given Mr. Hehner and himself (Mr. Richmond) the credit of being the first to calculate the total solids from the fat and the specific gravity; but they had only modified a formula which had been used before them by Fleischmann and Morgen.

Mr. Jones said that he had not seen the machine before. It was well known that the difference in the results obtained by the Adams process and by the old method of extraction from the solids was about 0.4. That had been accepted. The cause of the discrepancy was also well known, viz., that the fat was retained in the solids-not-fat. He could not explain the difference in this particular case. But if it was due to graduation,

he thought it could be got over satisfactorily. It was easy to rectify the graduation of the tubes, say by taking a larger quantity of milk.

Mr. Lister explained that the time mentioned for an experiment, viz., ten minutes, came from America. He had tested the Lister-Babcock machine and had obtained exactly similar results, whether he had turned for ten or for two or three minutes. The ten minutes rule had been strictly followed out here, perhaps because it had come from America.

Mr. Cassal said that at the time the paper, which had been alluded to on centrifugal separations, had appeared, he was himself engaged in making experiments in that direction, and the few results which had been obtained by Mr. Gerrans and himself, up to the time of publication of the paper referred to, had led them to hope that there was an important future for the centrifugal system of separating mixtures of liquids, and mixtures of liquids with solids, as in the case of precipitates. There was much to be done in that direction. In this respect he thought the machine before them might be very valuable.

Mr. Woosnam said that he believed it had been stated by Mr. Hehner that the red colours seen in the mixtures in the Babcock test-glasses was always obtained when an acid acted upon fats in the presence of a carbohydrate. He (Mr. Woosnam) was inclined to think, therefore, that the low results obtained by the Babcock process were due to loss of fat from the action of the strong acid, rather than to errors in graduation. He did not believe that this was a machine which analysts would take up for the purpose of testing milk (although they might possibly do so for throwing down precipitates) because there were other processes which were far more reliable and accurate. He considered that for dairymen, &c., the use of strong sulphuric acid was dangerous, and the machine for this reason unsuitable.

Dr. W. J. Sykes called attention to a recent extension of the use of the centrifugal machine. A paper had been read a short time since by Dr. Croll showing that it was possible in this way to entirely separate yeast and other matters suspended in beer, leaving the latter in an absolutely brilliant condition. The author of the paper thought that this might even be effected on a commercial scale.

Mr. J. Baynes then exhibited an apparatus for distilling off the ammonia in the Kjeldahl process for the estimation of nitrogen in organic substances. With this the proceedings of the Society terminated.

ON A SOURCE OF ERROR IN THE VOLUMETRIC ESTIMATION OF CHLORIDES BY MOHR'S METHOD.

By W. GATHORNE YOUNG.

(*Read at the Meeting, March 1st, 1893.*)

THE reliability of Mohr's method for the volumetric estimation of chlorides by titration with silver nitrate, in which chromic acid, or rather a neutral chromate, is used as an

indicator, has been questioned by Biscaro, whose observations have been confirmed by Forbes-Carpenter. These investigators attribute the error to the solubility of the neutral silver chromate in a solution containing nitrates, especially those of the alkalies. My father also has pointed out, in a paper read before this Society at one of its first meetings, the inaccuracy of this process when certain phosphates are present. During some experiments on chlorides I was led to believe that, although the presence of nitrates in large quantities may greatly influence the result, the most important considerations are the temperature and volume of the water present.

I therefore made several experiments on known quantities of NaCl dissolved in water at different temperatures and volume, the results of which show that in order to obtain correct estimations by this method, it is necessary to keep the volume of the solution very low, and to operate upon the smallest amount of chloride consistent with accuracy, in order that the volume of silver nitrate solution to be added may be small, as the water introduced in this way interferes considerably with an accurate determination as to the end of the reaction.

The sodium chloride solution used was made by dissolving 1 gramme of the pure re-crystallised salt in 250 c.c. of distilled water at 15.5°C. The silver nitrate was prepared from fresh crystals of the perfectly pure and dry salt by dissolving 4.79 grammes in 1 litre of water at 15.5°C., 1 c.c. of this solution being therefore equivalent to .001 gramme chlorine.

The first six experiments were made on 5 c.c. of the NaCl solution, that is .02 grm. NaCl; they were conducted at the normal temperature 15.5 deg. C. The results are as follows:—

First Series.

1.	5 c.c. NaCl solution evaporated to dryness required	12.2 c.c.
2.	5 c.c. " " " as before "	12.2 c.c.
3.	5 c.c. " " titrated without evaporation required	12.2 c.c.
4.	5 c.c. " " made up to 25 c.c. with water at 15.5°C. required	12.3 c.c.
5.	5 c.c. " " " " 50 c.c. required	12.4 c.c.
6.	5 c.c. " " " " 250 c.c. "	12.8 c.c.

As the volume of AgNO₃ solution which should be required for .02 grm. NaCl is 12.14 c.c., the error in the last experiment amounts to nearly 5 per cent.

From these results it will be seen that the most accurate determinations were obtained when the NaCl solution was evaporated to dryness and titrated in that condition, and that the error increases as the excess of water.

In the second series of experiments, 25 c.c. of the NaCl solution, which contains .1 grm. NaCl, were used, as it was thought advisable to try the effect on the result of larger quantity of the salt.

Second Series.

- | | | | |
|---|-----|-----|-----------|
| 1. 25 c.c. evaporated to dryness and titrated, required | ... | ... | 61 c.c. |
| 2. 25 c.c. evaporated to a small bulk (about 5 c.c.) required | ... | ... | 61 c.c. |
| 3. 25 c.c. evaporated to dryness and dissolved in 25 c.c. water, required | | | 61.2 c.c. |
| 4. 25 c.c. at 15.5° C., titrated without evaporation, required | ... | | 61.2 c.c. |
| 5. 25 c.c. made up to 250 c.c. with water at 15.5° C., required | ... | | 62 c.c. |

25 c.c. of the NaCl solution should require 60.7 c.c. AgNO₃ solution, so the same fact will be observed from these results, namely, that the error increases with dilution. It is interesting that although the amount of sodium chloride used, and therefore of sodium nitrate in the solution, was five times as much as in the first series, the nitrate present had no effect on the result.

An experiment was then made on 250 c.c. of pure distilled water, free from NH₃. This was titrated with the standard silver solution. There was no perceptible change in colour until .8 c.c. had been added, though, of course, the water was absolutely free from chlorides.

Two experiments were then made at 38° C. on 5 c.c. of the sodium chloride solution. In actual practice determinations are often made at about this temperature, as it is customary to wash residues, etc., with fairly warm water, in order to extract chlorides, and to titrate without cooling.

Third Series.

- | | | |
|---|-----|-----------|
| 1. 5 c.c. NaCl solution made up to 100 c.c. at 38° C., required | ... | 12.6 c.c. |
| 2. 5 c.c. " " " 250 c.c. " " | ... | 13 c.c. |

The volume of AgNO₃ solution, which should have been used, is 12.14 c.c.

From these results it will be seen that if it be necessary to dissolve the chloride to be estimated in even moderately warm water, it is very important that the solution be cooled before titration, as the presence of heat increases the error, and that in no case should the titration be performed on a very hot or boiling solution, as will be seen from the following experiments.

Fourth Series.

- | | |
|---|-----------|
| 1. 5 c.c. NaCl solution made up to 100 c.c.; titrated at 100° C. required | 13.1 c.c. |
| 2. 5 c.c. " " " 250 c.c.; " 100° C. " | 14.8 c.c. |

In both cases the volume of AgNO₃ solution taken should have been 12.14 c.c.; the error, therefore, in the last experiment is no less than 17 per cent.

As all these results tend to show that the error is caused by the silver chromate dissolving in the excess of water present, it was thought advisable to determine its solubility.

Some neutral silver chromate was prepared by decomposing potassium chromate with silver nitrate. The resulting precipitate was washed with boiling water repeatedly and thoroughly. It was then dried. About 2 grammes of this were, in order to effectually free the salt from any impurity, digested with boiling water for about 12 hours, more water being added from time to time as the solution evaporated. The undissolved chromate was allowed to settle down and the clear solution decanted. The chromate was again washed by decantation, about 300 c.c. of water added, and the whole digested on a sand-bath until a saturated solution was obtained; it was then set by for a day. The solution was then decanted, cooled to 15.5°C ., and the solubility of the chromate at that temperature ascertained by evaporating 100 c.c. to dryness in a carefully weighed platinum basin.

It was found that 100,000 parts of water at 15.5°C . dissolve 6 parts of chromate.

The solubility in boiling water was then determined with the result that 100,000 parts of boiling water dissolve 18 parts of chromate.

These experiments show that fairly accurate results may be obtained by Mohr's process, if the solution be kept down to a small bulk and at a low temperature.

This is rather important, as in many cases, more especially in water analysis, it is usual to take a certain volume of the solution for examination and to estimate the chlorides without previous concentration. The error thus obtained is considerable; as, in the drinking water supplied to London, the solution of chlorides must be very dilute as the chlorine seldom exceeds .02 grammes per litre.

In order to show the importance of these errors in the case of water analysis, I made the following experiments on East London water:—

Fifth Series.

1. 100 c.c. evaporated to dryness and dissolved in about 2 c.c. water, gave 1.26 grains Cl per gallon.

2. 100 c.c. titrated at once, cold, gave 1.47 grains Cl per gallon.

3. 100 c.c. „ 38°C . „ 1.61 „ „

4. 100 c.c. „ 190°C . „ 1.82 „ „

These experiments were repeated with precisely similar results.

In the last case the error is nearly 31 per cent. In the experiments in which the water was first evaporated and then dissolved in a small quantity of water, the titrations were performed in the platinum basin, in which the evaporation was made.

It may be mentioned that in the experiments on dilute heated solutions, though the error shown is very great, it would be even greater, had the appearance of a permanent red precipitate been taken as indicating the end of the reaction.

In these cases the change of colour was so very gradual that I found it extremely difficult to decide unless the solution were compared with a very dilute solution of potassium chromate, and the slightest change in tint taken as the end.

If after this more silver nitrate be added, no precipitate appears for some time, the only change being a deeper red colour to the solution owing to neutral silver chromate Ag_2CrO_4 going into solution.

In estimating chlorides with standard silver nitrate, therefore, it is advisable to evaporate to dryness before titration and weak solutions of silver nitrate are to be condemned when working upon anything but very small quantities of chloride, on account of the dilution which they cause.

DISCUSSION.

The Chairman welcomed Mr. W. G. Young as the son of an old and valued member of the Society, and invited discussion on the Paper just read.

Mr. R. Bodmer presumed that the chromate of potassium used in the experiments was free from chlorides. It was a good plan to have by the side of the basin in which the determination was made a similar basin containing pure distilled water coloured with the same amount of the chromate solution. The reading was thereby rendered much finer. The extreme differences recorded by Mr. Young with the same solution were certainly surprising.

Mr. H. Droop Richmond asked Mr. Young if he had noticed in *THE ANALYST** three or four years ago, a paper abstracted from the *American Chemical Journal*, in which very similar results to those he had put on the board were given. He (Mr. Richmond) did not think that the experiments recorded in the paper he referred to treated of the influence of temperature; but they showed that the error increased with the bulk of the solution, and a correction formula was given. He would like to know if Mr. Young had consulted that paper, and, if so, how the results compared with his own.

The Chairman asked Mr. Young if he had tried the method which he (Dr. Dupré) had proposed some years ago,† of conducting the process, with potassium chromate as an indicator, in a porcelain dish, and viewing the sodium chloride solution through a solution of potassium chromate solution of approximately the same tint as the contents of the dish. The first faint appearance of a red colouration is in this way rendered much more distinct. The marked increase of delicacy in using the process by gaslight had led him to adopt this method in daylight.

Mr. Young, in reply, said he had not seen the paper referred to by Mr. Richmond. He had gone through a good many of the later Journals of the Chemical Society to search for any paper on the subject, but had failed to find any. He had asked the

* *THE ANALYST*, vol. xiv., 229.

† *THE ANALYST*, vol. v., 123.

opinion of two or three well-known chemists—among them Professor Ramsay—and they had said that the solubility of chromate of silver was utterly unknown to them. He had found difficulty in making theoretically correct standard solutions. He had made up the solution for this work with the purest silver nitrate he could get, and had found a great difficulty in standardizing it. He thought that the error was due to the solubility of the chromate of silver. He had not used a vessel filled with a solution of potassium chromate such as Dr. Dupré had described. He quite agreed with Dr. Dupré that by using such an arrangement the difficulty in accurately observing the change of colour would be got over.

Dr. Dupré asked Mr. Young if he had standardized against salt.

Mr. Young replied that he had found it impossible to standardize accurately. He titrated his silver nitrate solution against a known quantity of sodium chloride, and the silver solution appeared to be very slightly weak. As there could be no doubt about the purity of the silver nitrate, he had adopted this solution as correct. The strength of the solution would not affect the differences in the results.

Mr. Bodmer asked the author, Mr. Young, whether the nitrate of silver used was the fused or the crystallized nitrate.

Mr. Young replied that he had used crystallized silver nitrate, and that he had recrystallized it twice himself.

Mr. Bodmer stated that if ordinary commercial common salt were used the same results were not arrived at as when rock salt was used. Absolutely correct results could be obtained by using fused nitrate of silver.

THE LEFFMANN-BEAM METHOD FOR THE ESTIMATION OF FAT IN MILK.—PART II.

By H. DROOP RICHMOND.

(Read at the Meeting, December 7th, 1892.)

In the former portion of this paper (*THE ANALYST*, xvii. 144), I showed that the volume of fat depended on an extension of Henry's law, and I showed that this held with considerable exactitude; my experiments, however, were conducted using a set of bottles made by Messrs. Müller and Co., which were graduated so that 86 divisions equalled 1.475 c.c., and occupied a length of 5.3 centimetres, and the capacity varied from 28.5 c.c. to 30.5 c.c.; this variation I found to make no difference. I have since experimented with a new series of bottles, obtained from Messrs. Baird and Tatlock; these were graduated so that 100 divisions equalled 1.875 c.c., and occupied a length of 5.53 centimetres, and the capacity was 29 c.c.

With these latter I find that the same law does not hold; these bottles had been graduated on the assumption that, using the proportions of milk, fusel mixture, and acid

given by me, 80 divisions ($= \frac{80}{1.07}$) should occupy 1.5 c.c.; this would have been substantially correct had the extension of Henry's law given by me been applicable to conditions other than those under which I worked, but it evidently is not. Before entering into the theory of this subject, I propose to give a few results with the bottles received from Messrs. Baird and Tatlock, the English agents for this machine. I have found it necessary to multiply the readings by 1.08 in order to obtain the actual percentage of fat in the milk; with this correction the results are as accurate and reliable as I had previously found. In fact, the more I have used this method the more I have been convinced of its accuracy, and this, combined with the speed and ease of working, places it in the front rank of practical analytical methods.

SERIES IV.

No.	Amount of Fat.	L. and B.	L. and B. corrected.	Error.
68	... 3.98	3.7	4.00	+ .02
69	... 3.65	3.35	3.62	— .03
70	... 4.64	4.2	4.54	— .10
71	... 2.93	2.7	2.92	— .01
72	... 4.05	3.75	4.05	—
73	... 1.96	1.8	1.94	— .02
74	... 3.87	3.6	3.89	+ .02
75	... 4.24	3.9	4.21	— .03
76	... 3.62	3.45	3.73	+ .11
77	... 3.60	3.3	3.56	— .04
78	... 3.56	3.3	3.56	—
79	... 3.58	3.3	3.56	— .02

These results are but a few of those actually obtained, and are not in any way picked, but are extracted at random from my note books.

To turn to the theory of the subject; it was at first startling to find that a comparatively small variation of the conditions, i.e., the size of bottles, should make such a large variation in the results, indicating that the theoretical considerations I had adopted were not wholly correct. I therefore, in order to test these, made the following experiment:—

80. About 15 grams of the fatty layers obtained in various estimations were shaken with boiling water until the water was practically neutral; this was filtered and neutralized with decinormal barium hydrate, a large precipitate separating; the solution was filtered and washed with hot water; the filtrate was found only to contain small quantities of barium, and a very notable proportion of chlorine, so that the presence of considerable quantities of amyl-hydrogen sulphate in the fatty layer was disproved, and my assumption that the layer measured contained this, was not correct.

Were there nothing to increase the volume of the fat the amount equalling 8.0 per cent. fat would be 1.35 c.c., while in B. and T.'s bottles it is 1.39 c.c., and in M. and Co.'s it is 1.475. To what is the excess due?

While drawing off the fatty layers I was struck by the way in which the fat adhered to the sides of the dry tube used to remove it, while it did not adhere to the neck of the flask. This suggested to me that there was a layer of aqueous liquid between the fatty layer and the sides of the neck, and consequently that the actual diameter of the fatty layer was less than the internal diameter of the neck. To enter deeper into this subject, which involves the consideration of difference of surface tension between three substances—the fatty layer, the acid mixture, and the glass, and of capillary attraction, is impossible with the meagre data that exist; the very fact of the fat rising so completely and rapidly shows that the differences of surface tension must be enormous, and play a very important part in the solution of this question.

I must, therefore, renounce the theory that amyl-hydrogen sulphate is dissolved in the fat, and that this accounts for the increase of volume. It is not, therefore, necessary to employ the extension of Henry's law given in Part I. My results emphasise the fact that this method is essentially a laboratory method; the actual factor connecting readings and percentages of fat must be worked out for each bottle, and it will not do to trust to the graduations, even if the absolute volume is checked. As I find that my two series of bottles agree among themselves, I have hoped that by adopting fixed dimensions, bottles can be graduated in quantity to read percentages; but it is possible that one quality of glass must be adhered to. There is some evidence that the quality of glass does not make very much difference, as I have obtained substantially the same results with B. and T.'s bottles as with a bottle made by myself, using the broken neck of one of the bottles received from Messrs. Leffmann and Beam. The diameter of the two necks differs only by 0.02 mm.

I prefer the size of neck adopted by Müller & Co. of internal diameter 0.5955 c.m. as giving larger divisions and more exact readings, and I would suggest that bottles should be made with necks of this diameter, and graduated so that 80 divisions = 1.475 c.c. and occupy a length of 5.3 centimetres, that the volume should be 29 c.c., and that the same glass should be used for all.

Analysis of Cream.

I have made a number of experiments on the analysis of cream, and find that the measurement of the cream in a 10 c.c. specific gravity bottle is not sufficiently exact for creams containing more than 30 per cent. of fat. It is necessary to weigh it; for this purpose I measure a definite volume of cream, 3 c.c. if using B. and T.'s bottles, and 2.5 c.c. if using M. & Co.'s, into the previously-weighed bottles, and weigh it. I then add 12.2 or 12.7 c.c. of water respectively, and fusel mixture and sulphuric acid as usual,

The reading is multiplied by 15.74 for B. and T.'s bottles, and 14.11 ($= \frac{15.4}{1.07}$) for M. and Co.'s bottles, and divided by the weight of cream taken; the results are only accurate to about 1 per cent.; this, however, is sufficient for the ordinary purposes of cream control.

No.		Fat Gravimetric.	L. & B.	Error.
81.	...	50.9 W.S.	50.5	— .4
82.	...	42.8 V.	43.0	+ .2
83.	...	25.6 W.S.	24.3	— 1.3
84.	...	48.9 W.S.	49.7	+ .8
85.	...	44.3 V.	44.1	— .2
86.	...	45.9 W.S.	46.2	+ .3
87.	...	47.9 V.	48.0	+ .1
88.	...	46.3 V.	47.3	+ 1.0

The above were obtained with Müller & Co.'s bottles.

No.		Fat Gravimetric.	L. and B.	Error.
89.	...	40.1 W.	39.2	— .9
90.	...	41.9 W.	41.2	— .7
91.	...	47.0 W.	48.5	+ 1.5
92.	...	37.9 V.	38.4	+ .5
93.	...	45.0 V.	44.5	— .5

These were obtained using B. and T.'s bottles.

Nos. 92 and 93 were each done in triplicate to test the obtainable accuracy.

The results were:—

No.				
92.	...	38.8	38.1	38.3
93.	...	45.2	43.9	44.4

I have found that with M. and Co.'s bottles, the weight of fat deduced from the results obtained with milk, can be used when analysing cream, while with B. and T.'s bottles it is necessary to divide it by 1.06.

Sundry Experiments on Milk.

A few experiments were made using both sets of bottles:—

No.		M. and Co.	M. & Co. $\times \frac{1}{1.07}$	B. and T.	B. & T. $\times 1.08$
94.	...	4.25	3.98	3.7	4.00
95.	...	4.36	4.03	3.75	4.05
96.	...	3.8	3.55	3.3	3.56

Another set of experiments was made comparing the results obtained by using the fusel oil mixed with sulphuric acid, with those obtained in the ordinary way.

No.	Sulphuric Acid.			Hydrochloric Acid.		
97.	3.56	3.56
98.	3.56	3.56
99.	3.50	3.56

In order to see how the process agreed with itself when the results were repeated, six bottles were all filled with the same milk (B. and T.'s bottles being used); the results were:—

No.	...	Reading	3.8	—	3.8	—	3.75	—	3.8	—	3.8	—	3.8
100.	...	Fat	4.10	—	4.10	—	4.05	—	4.10	—	4.10	—	4.10

Conclusions.

The results recorded in this paper show—

(i.) That this method is very accurate and reliable when once the bottles are graduated.

(ii.) That it is necessary to standardize each bottle before use; this shows that it is unsuited for use outside the laboratory, unless the bottles are first tested.

(iii.) That creams may be roughly analysed by its means; the analysis of cream by this process is not so satisfactory as the analysis of milk.

(iv.) That the use of hydrochloric acid is not absolutely necessary; the fusel oil may be mixed direct with the sulphuric acid; the separation of the fat is not quite so sharp in this case, and, on this account, the omission of the hydrochloric acid is not to be recommended.

The principle of this method appears to be quite distinct from that of any other process of fat estimation. Besides the principle of dissolving the albuminoid employed in the methods of Hoppe-Seyler, Soxhlet, Röse, Planchon, Cochran, and last, but not least, Werner-Schmid, and the application of centrifugal force, and measuring of the fat due to Babcock, there is the use of fusel oil to promote by a large difference of surface-tension the easy separation of the fat. This places the Leffmann-Beam method in a distinct category, and renders it the most perfect of volumetric processes for the estimation of fat in milk.

I have to record my thanks to Mr. A. Schnepel for help in this investigation.

The Volatile Fatty Acids of Butter. H. Kreit. (*Schweiz. Wochenschr. Chem. Pharm.*, 1892, xxx., 449, through *Chem. Zeit.*)—The author confirms the statement of M. Schrodtt and O. Henzold that the Reichert-Meissl figure is dependent on the period of lactation. Of 75 samples examined during a year, the Reichert-Meissl figure ranged from 21.1 to 34.4; 4 per cent. were under 22; 24 per cent. between 22 and 24; 33 per

cent. between 24 and 26; 23 per cent. between 26 and 30; and 17 per cent. over 30. All the samples that gave a figure higher than 30 were from cows which had recently calved.

B. B.

Boric Acid as a Normal Constituent of Beer and a Natural Constituent of Hops. J. Brand. (*Zeits. gesamt. Brauw.*, 1892, xv., 427, through *Chem. Zeit.*)—The method of detecting the presence of boric acid used by the author was that depending upon the well-known reaction with turmeric paper. The process adopted consisted in making the beer to be examined slightly alkaline, evaporating it to dryness and incinerating, extracting the ash with water, evaporating the solution nearly to dryness, making faintly acid with hydrochloric acid, and heating on the water-bath after the immersion of a strip of turmeric paper. Beer containing only 0.1 per cent. of boric acid gave a definite reaction under these conditions, and the turmeric paper afterwards afforded the characteristic green coloration with alkali. Pure beer, to which, according to the author, no boric acid had been added in the course of manufacture, also gave the reaction. Beer of various origins was examined, the samples being Munich, Bavarian, German, Austrian, and Brazilian, and boric acid was found in all. In order to put the presence of boric acid beyond doubt, Gooch's method, depending on the volatility of methyl borate, was used. The ash from 200 c.c. of beer was distilled with pure methyl alcohol and sulphuric acid, and the distillate received in ammonium carbonate solution. The liquid in the receiver was evaporated to a small bulk, supersaturated with hydrochloric acid, a strip of turmeric paper dipped in, and the whole taken to dryness. Boric acid was recognised in all cases. The origin of the boric acid now engaged attention. Various samples of the ash of malt and barley were examined, but no trace of boric acid found. The ash of hops, however, gave marked indications of boric acid. Eighteen different kinds of hops were examined, 5 grams of each being incinerated without the addition of alkali, and the ash distilled with methyl alcohol and sulphuric acid, and in all cases turmeric paper was coloured and gave the usual subsequent reaction with alkali. It therefore appears that the boric acid normally present in beer is due to that existing as a natural constituent of hops. Hops, both cultivated and wild, plucked direct from the growing plant, were found to contain boric acid, which was detected in the leaves, stems, and tendrils, as well as in the actual flower.

B. B.

Ammonium Thiocyanate in Manures. P. L. Jumeau. (*J. Pharm. Chim.*, 1893, xxvii., 190, through *Chem. Zeit.*)—The author, in examining a sample of nitrogenous manure and one of ammonium sulphate, which proved deleterious to the crops for which they were used, found that they contained ammonium thiocyanate, which was the cause of the evil effects observed. The sample of ammonium sulphate had the following composition:—Moisture, 10.71; ammonium sulphate, 67.84; ammonium thiocyanate, 9.39;

sodium sulphate, 9.24; potassium sulphate, 0.98; calcium sulphate, 0.68; ferric oxide, 0.30; silica, 0.08 parts per cent. respectively, and traces of chlorine and magnesia. The determination of the thiocyanate was effected by dissolving 5 grms. of the sample in 200 c.c. of water, filtering, and determining the sulphate in 10 c.c. of the filtrate. Another portion of 10 c.c. was acidified with hydrochloric acid, the thiocyanate oxidised with potassium permanganate, and the resulting sulphate determined by precipitation with barium chloride in the usual way. The difference between the two determinations of sulphate gave a measure of the amount of thiocyanate present. An alternative method, which is available in the absence of chlorides, consists simply in titrating the aqueous solution with silver nitrate in the presence of a small quantity of nitric acid. The addition of a little ferric chloride serves to provide an indicator, the reaction being completed when the liquid is decolourised. The procedure can, of course, be modified by adding an excess of silver and titrating back with a standard solution of ammonium thiocyanate. Should chlorides and cyanides be present, the following plan may be adopted:—A standard solution of ammonium thiocyanate is prepared, its value being accurately determined by means of titration with silver nitrate or by oxidising with permanganate and weighing the resulting sulphate as barium sulphate. It should have a strength of about 8 grammes per litre. A solution of potassium permanganate is then prepared, containing about 10 grammes per litre, and used to titrate the standard solution of ammonium thiocyanate, of which 10 c.c. are taken, diluted to 100 c.c., and acidified with 10 c.c. of pure sulphuric acid. The appearance of a permanent pink colour indicates the end of the reaction with perfect sharpness. The same process is then used for the sample to be analysed, of which about 0.5 grammes is taken. It is alleged that the permanganate has no action on cyanides and chlorides under these conditions, a statement that seems in need of confirmation.

B. B.

Methyl and Ethyl Alcohols. C. A. Lobry de Bruyn. (*Bericht. deutsch. Chem. Gesell.*, 1892, xxvi., 268–274.)—The author shows that the solubilities of ammonia, and of potassium cyanide, potassium iodide, mercuric cyanide, and hydroxylamine hydrochloride are greater in methyl than in ethyl alcohol. Colourless solutions of sodium methylate, containing 0.1 to 0.2 gramme Na per c.c. can be prepared from methyl alcohol, care being taken that during the addition of the sodium they are kept cool. Such solutions, when kept out of contact with cork and similar organic substances, do not become brown, and when preserved in a dry atmosphere, free from carbonic anhydride, do not alter in titre. Solutions, either of sodium or soda, in methyl alcohol are, therefore, to be preferred to those in ethyl alcohol for such uses as that of the titration of free fatty acids in fats. Sodium ethylate is much more readily oxidised than sodium methylate; consequently, the former possesses stronger reducing properties than the latter. Spongy platinum and potassium permanganate act on methyl alcohol much less readily than on

ethyl alcohol, and dry bleaching powder, which is without action on the former liquid at the ordinary temperature, spontaneously interacts with the latter in from 7 to 10 minutes. When chlorine is passed into either of the alcohols a reaction sets in much sooner in the case of ethyl than in that of methyl alcohol. The difference in reactive power is still more marked in the case of bromine. A mixture of bromine and methyl alcohol, containing 61.7 per cent. of bromine was still found to contain 57 per cent. of bromine after 78 days. Iodine dissolves in the two pure alcohols to about the same extent; but whereas a saturated solution of iodine in methyl alcohol does not alter its titre after many weeks, one in ethyl alcohol gradually becomes weaker. The apparent larger solubility of iodine when it is triturated with ethyl alcohol than when it is left at rest with this liquid, is due to the fact that the reaction products are better solvents for iodine than is the pure alcohol.

Methyl alcohol is somewhat more hygroscopic than ethyl alcohol.

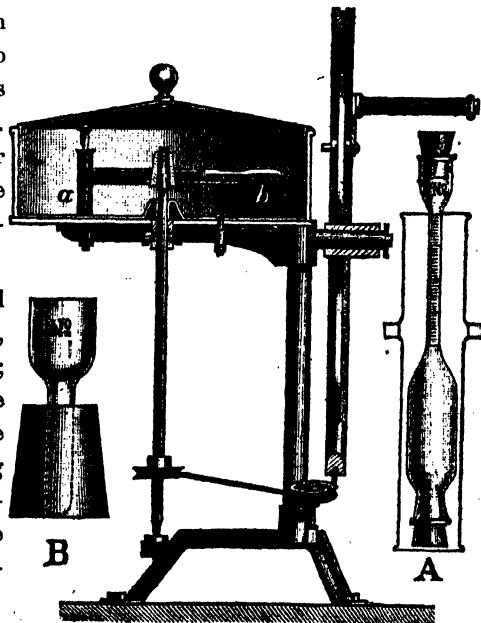
A. R. L.

Acido-butyrometry. N. Gerber. (*L'Industrie Laitière*, 1892, 50, 397; 52, 413; 1893, 1, 1; 6, 43.) The author, dissatisfied with the methods for the rapid estimation of fat in dairy products, has devised a new method for the purpose. The proposed method is essentially the same as that due to Leffmann and Beam, insomuch that amyl alcohol is employed to promote the easy separation of the fat from the aqueous portion.

The centrifugal machine used is shown in the figure, in which the bottles are also figured. These are of two kinds, for liquids and for solids, such as cheese and butter. The one shown in the figure is that used for solids; that used for liquids differs from the other by having the top closed. The estimation of fat is performed as follows:—

Milk.—10 c.c. of milk are measured into the bottle, 1 c.c. of amyl alcohol is added, and then 10 c.c. of strong sulphuric acid; a cork is inserted into the open end, the mixture shaken and whirled in the machine for 2 to 2½ minutes. During the whirling the outer case of the machine should contain water kept warm by a spirit-lamp; the machine is allowed to run down, and the percentage of fat is read directly.

Cream, butter, cream-cheese (*fromage à pâte molle*).—1 c.c. is measured in the cup B (see figure), which is fixed firmly into a cork, which goes into the lower end of the



bottle; 8 c.c. of water are added, and amyl alcohol and acid as before. The reading must, in this case, be multiplied by 10 to obtain percentages. If great precision is required, or if ordinary cheeses are to be analysed, 1 gramme should be weighed instead of 1 c.c. being measured; but the author is of opinion that measurement is sufficiently exact in practice.

H. D. R.

Rapid Determination of Crude Fibre by Means of the Centrifugal Separator. W. Thörner. (*Chem. Zeit.*, 1893, xvii. 394 to 395.)—The tedious process of determining crude fibre in food stuffs is much expedited by the aid of the centrifugal methods already described by the author for various other analytical separations (*THE ANALYST*, 1891, 210). The vessel used is a tube with a collar or ridge near the mouth, so as to allow it to hang in the water-bath during the solution of the material to be analysed. It has a capacity of 50 c.c., and the quantity of substance taken for analysis is 1 gramme. Should the food stuff, in which the crude fibre is to be determined, contain any considerable quantity of fat, it is first treated with 20 c.c. of ether, and the tube containing it rotated for one or two minutes in the machine, after which the ether can be poured off without fear of loss, and the operation repeated once or twice until the fat is completely removed. The ether is then driven off the residue by heating in the water-bath, 30 c.c. of hot water added, and the heating continued for about 10 minutes, the contents of the tube being stirred with a glass rod provided with a flattened knob at the end. 10 c.c. of dilute sulphuric acid are added (50 c.c. of the strong acid made up to a litre), and the heating and stirring continued for another 30 minutes. The rod is withdrawn, the tube put in the centrifugal machine and rotated therein at a speed of 3,000 revolutions per minute, for 3 or 4 minutes. The insoluble matter is nearly completely separated in a compact form at the bottom of the tube by this treatment, and the more or less turbid liquid is poured off through a weighed filter sufficiently large to hold the whole of the contents of the tube. The tube is then refilled with about 40 c.c. of hot distilled water, and the stirring with the glass rod repeated for a period of 10 to 15 minutes, the tube being meanwhile suspended in the water-bath. The clear liquid is decanted through the filter and the washing repeated. The residue in the tube is then treated with 30 c.c. of hot water and 10 c.c. of a solution of caustic potash containing 50 grammes of KOH per litre, heated in the water-bath, and stirred as before for half-an-hour. Washing with water, as already described for the treatment with acid, is then performed, and the crude fibre thrown on to the filter, washed in succession with alcohol and ether, dried and weighed in the usual way. The whole determination can be completed in three or four hours, and with a moderate-sized centrifugal machine 8 determinations can be carried on simultaneously.

B. B.

REVIEWS.

DIE CHEMISCHEN UNTERSCHIEDE ZWISCHEN KUH UND FRAUENMILCH UND DIE MITTEL ZU IHREER AUSGLEICHUNG. (The chemical differences between human and cow's milk and the means of compensating them,) F. Soxhlet. Pamphlet, J. F. Lehmann, Munich, 1893. Price 60 pf. (7½d.)

The author shows that the differences between the two milks are :—

1. The different behaviour of the casein when coagulated. 2. The different amount of salts. 3. The difference in the amount of total food-material and in the relative proportion of the constituents.

As dilution modifies the behaviour of the casein of cow's milk towards rennet-enzymes and brings it nearer to the casein of human milk, he proposes to make an artificial human milk by diluting cow's milk with half its volume of a 12·3* per cent. solution of milk sugar.

The composition of human milk, cow's milk, and artificial milk are said to be :—

	Human	Cow	Artificial
Water	87·41	87·17	85·30
Albuminoids	2·29	3·55	2·37
Fat	3·78	3·69	2·46
Milk Sugar.. ..	6·21	4·88	9·40
Ash	0·31	0·71	0·47

The seeming excess of milk sugar is due to an allowance of 2·43 parts of sugar for each 1 part deficient of fat, after the observations of Rubners; to meet the deficiency of 1·32 of fat, 3·19 of milk sugar has been added. Professor Soxhlet takes no notice of Frankland's principle of preparing artificial human milk. He condemns the addition of cream to make up the deficiency of fat, on account of there being no method of estimating the fat in the materials used; he considers his preparation to be capable of giving a milk of constant composition, and even states that the milk of normally fed cows is of constant known composition. How erroneous his view is will be evident to our readers; the rapid and accurate methods of fat estimation and the variations in the composition of cow's milk are too well known to need a reference here. There seems no justification for, to quote Soxhlet's own words, "adopting the less correct plan of making up the deficiency of fat by some substance as nearly as possible equivalent to fat," and to substitute a rough estimate of the food-value of milk sugar for an accurate estimation of fat.

* Note by Reviewer.—Though Soxhlet directs the use of half the volume of a 12·3 per cent. solution of milk sugar, a simple calculation shows that a solution containing 18·44 per cent. of anhydrous milk sugar, or 19·4 per cent. of hydrated sugar must be used to obtain the composition given by him; such a solution is nearly saturated and can only be prepared by long agitation in the cold or by means of heat. There are other arithmetical errors.—H. D. R.

Professor Soxhlet reviews the evidence available as to whether milk sugar can be replaced in infants' foods by other bodies of a similar nature, *e.g.*, cane sugar, dextrose, and maltose, and very properly comes to the conclusion that it cannot be so replaced; milk sugar has dietetic properties not possessed by any other available sugar.

With the exception of the portion alluded to, to which objection may be taken, this is a most valuable contribution to our knowledge of milk and the pamphlet will well repay perusal.

H. D. R.

GALENIC PHARMACY. By R. A. Cripps, F.I.C., Pharmaceutical Chemist. (London, J. & A. Churchill.)

This work ably fills a void which has hitherto existed in modern literature, for, as the author says in his preface, "while works on Chemistry, Botany, and Physics have amply provided the student with instruction in these sciences, he has been left almost without help in their application to Pharmacy."

After an introduction, the author devotes a chapter to preliminary chemical notes, in which he explains, somewhat unnecessarily we think, the use of symbols and formulæ and gives definitions of elements, compounds, atoms, molecules, &c. The chapter which follows on the structure of plants is more in place and the descriptions are concise and clear.

The author then proceeds to discuss Pharmacy proper, describing in detail, with the aid of excellent wood-cuts, the various processes required to be carried out in a Pharmaceutical Laboratory. Trituration, Solution, Filtration, Evaporation, Distillation, &c., are all well and fully described. Where definite chemical reactions occur the formulæ are given, and the various tests for the preparations are detailed.

The work, which seems admirably adapted for its intended purpose, concludes with a number of useful definitions, and an appendix containing Tables of Temperature, Specific Gravity of Spirit, and other useful data.

Throughout the book there is abundant evidence of practical knowledge of the subjects under treatment, and one is forcibly struck with the contrast between the author's able descriptions and the imperfections of that monument of slovenly and perfunctory work, the British Pharmacopœia.

The work is a credit to the author, who is both an able Chemist and a well-known Pharmacist, and will be found very useful to Public Analysts, who are rather apt to give less attention to that portion of their duties bearing on Pharmacy, and especially Galenic Preparations, than is desirable.

The whole work covers some 320 pages and should find a place in the laboratory of every practising analyst.

A. H. A.

THE ANALYST.

JUNE, 1893.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on the 3rd May, at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Alfred H. Allen occupied the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed:—As Members, Mr. George H. Allibon, Belfast; Mr. Anthony Roche, Dublin; Dr. H. J. Alford, Taunton. As Associates, Mr. Chas. Harrison, Mr. G. E. Scott Smith, Mr. L. K. Boseley.

The following gentlemen were duly elected:—As Members of the Society, Mr. William James Orsman, Wigan; Mr. Charles E. Sohn, Anerley. As Associates, Mr. A. H. M. Muter; Mr. C. Hubert Pope.

The following papers were then read by Mr. Boseley:—

THE ACTION OF HEAT ON MILK.

BY H. DROOP RICHMOND AND L. K. BOSELEY.

THE action of heat on milk has been studied chiefly regarding the changes in the albuminoids (c.f. Faber, ANALYST, xiv., 141). In a paper by one of us (ANALYST, xvii., 225) it is stated that the specific rotary power of milk sugar is reduced by heating.

The following figures obtained on milks heated to a temperature of 100° C. (boiling water) in closed vessels for different lengths of time will show the extent of reduction. The time of heating, unless stated, was about two hours, or, more exactly, it varied from 1½ to 3 hours. We had intended to heat milk for different times in order to see if we

could find a constant variation for a unit of time; but the results of Nos. 5 and 6 show conclusively that three samples heated for the same time may show enormous variations.

Milk Sugar Pol.	1.	2.	3.	4.	5.	6.	7.
Original	5.09	5.20	5.10	5.26	5.09	4.75	5.30
Heated about 2 hours	4.54	4.56	4.39	4.66	{ 3.88 4.40 4.37 }	{ 3.72 3.64 3.98 }	4.84
			4 hr.	4.24		$\frac{1}{4}$ -hr.	5.20

We have quoted the bulk of the results for two hours' heating and not for less times, simply to show the change on heating in a distinct manner. The numerous methods of milk sterilisation by heating will all produce the same change, though possibly to a different degree. A figure is given for $\frac{1}{4}$ -hour in No. 7, as the results there are means of about 50 polariscope readings, and are probably accurate. This shows that even in $\frac{1}{4}$ -hour the change may be detected by a delicate polariscope.

To show that the reducing power on Fehling's solution is constant, we may quote the following experiments:—

Original	5.31 (grav.)	4.41 (pol.)	4.92 (grav.)
Heated 2 hours ...	5.22 (grav.)	4.56 grav.	4.45 grav.
			4.88 (grav.)

It is well known that milk sugar has, besides its normal and bi-rotation, a half rotation (c.f. Vieth, *ANALYST*, xi., 141); we thought it possible that, on heating, a quantity of the half-rotating modification might be formed: Schulze and Tollens (*Annalen*, 271, 49) have shown that sugars dissolved in dilute ammonia always exhibit the normal rotation. We found, however, that no change was produced by adding ammonia, nor by allowing our solutions to stand some hours. It is noticed that when milk is heated for a long time to 100°, a brown colour is produced, and we have found that the rotation varies to some extent inversely as the colour. This is probably due to the formation of a "caramel," and it is well known that "caramel" prepared by heating dry sugar to 170° is inactive. Incidentally we may mention that in a sample of milk heated to 85° for a few minutes the albumen found by Sebelein's method was 0.19%.

Milk sugar in solution as in milk differs from dry milk sugar as it is caramelized at 100°. Weisberg (*Chem. Centr.*, 1892, ii., 458) has noticed a decrease in the rotary power of cane sugar in solution by several hours' heating.

THE BOILING POINTS OF SALT SOLUTIONS.

BY H. DROOP RICHMOND.

WISHING to find a convenient method of keeping a water bath at a constant temperature a little above 100° C., it struck me that by adding a proportion of salt (sodium chloride) to the water inside and keeping the level constant by the automatic addition of water, the desired effect might be obtained.

I found, however, that the temperature attained did not agree with that according to Legrand's tables (*Ann. Chim. Phys.*, 1835 [2], lix., 423) and therefore redetermined them. My apparatus consisted of a flask with a layer of pumice stone at the bottom, containing a considerable quantity (100 c.c.) of salt solution; through the cork passed a 5 c.c. pipette and a tube with a side tube attached, through which the thermometer was passed; the end of the pipette and the bulb of the thermometer were in close proximity.

The mode of operation was as follows:—The solution was boiled, and the thermometer was read at any point; immediately the pipette was washed out with the solution two or three times, and the thermometer again read to one-tenth degree; 5 c.c. were then withdrawn and placed in a stoppered weighing bottle, and the thermometer again read; the mean of the readings was taken as the temperature at the time of withdrawal of the solution. After cooling, a portion of the solution was weighed out into a 500 c.c. flask, and 100 c.c. were titrated with silver solution, with potassium chromate as indicator, care being taken that about 45 c.c. of silver solution were required in each case; the silver solution was standardised on such quantities of pure sodium chloride as took 45 c.c. of solution. The percentage of sodium chloride was calculated from the data obtained in each titration. The thermometer readings were corrected to 760 m.m. by taking the boiling point of water at the same time; as the stem was at 100° C. the correction for the mercury therein was very small.

The molecular percentages of sodium chloride were also calculated.

No.		Boiling point.	NaCl. %.	NaCl. Mol. %.	B. P. (calc.)
1	...	102.2°	7.6	2.47	102.15°
2	...	103.0°	11.0	3.63	103.16°
3	...	104.2°	14.9	5.11	104.44°
4	...	104.8°	16.1	5.58	104.85°
5	...	106.1°	18.8	6.66	105.79°
6	...	107.1°	22.3	8.12	107.06°
7	...	107.7°	24.0	8.87	107.71°
8	...	108.7°	26.0	9.76	108.49°
9	...	109.5°	28.7	11.04	109.61° (saturated solution)

My results are about 1° C. higher than Legrand's; the boiling point appears to be a direct function of the molecular percentage, and the boiling points calculated by adding the molecular percentage divided by 1.15 to 100° C. are within the limits of reading of the thermometer used. It is interesting to note that a saturated solution has nearly the composition $\text{NaCl} \cdot 80\text{H}_2\text{O}$. If time permits, I hope to investigate the boiling points of other salts to see if it is a general law that the boiling points are direct functions of the molecular composition of the solution, and also to determine the densities at the boiling point. If, however, any other observer having greater facilities than I have will do this, I shall be only too pleased to leave the work to him; a large and reliable series of determinations would be extremely valuable.

Wüllner (*Ann. Chim. Phys.*, 190, 564) finds the boiling point to vary as the amount of salt dissolved. Nicol (*Phil. Mag.* 22, 502) has attacked the question from a different point of view, and his conclusions do not apply to boiling solutions.

Papers were then read, entitled "Points in the Analysis of Condensed Milk," and "Note on the Detection of the Adulteration of Fresh Milk by Diluted Condensed Milk," both by Messrs. Richmond and Boseley.

(*Conclusion of the Society's Proceedings.*)

The Determination of Nitrogen in Nitrates by Reduction. T. F. Schmitt. (*Chem. Zeit.*, 1893, xvii, 173.)—The method is a modification of that already described by the author (*THE ANALYST*, 1893, 92), in which a mixture of powdered zinc and iron is used as a reducing agent. The process is carried out as follows:—10 grms. of the nitrate are dissolved and made up to 500 c.c. 10 c.c. of glacial acetic acid and 10 grms. of the metallic powder are placed in a flask of a capacity of about 750 c.c., and 25 c.c. of the solution of the nitrate added. The flask is covered during the reduction to prevent loss by spraying, and after solution is complete (which is the case in about ten minutes) the contents of the flask is diluted with 200 to 300 c.c. of water, 30 c.c. of caustic soda of sp. gr. 1.25 added, and the whole distilled as in the Kjeldahl process. It must be noted that it is essential that the iron be finely divided; it is mixed with the powdered zinc in equal parts. The total nitrogen can be determined in guanos and nitrate mixtures by the following simple alteration in procedure:—1 gm. of the substance is dissolved in water, 5 c.c. of glacial acetic acid and 2 to 3 grms. of the metallic powder added, and the whole gently heated for ten or fifteen minutes. After the contents of the flask have cooled, 25 c.c. of sulphuric acid are cautiously added in small portions, undue frothing being restrained by the addition of a fragment of paraffin wax. The acetic acid is then driven off by heating, and the remaining contents of the flask heated until the organic matter is completely decomposed, as in the Kjeldahl process, which happens in about two hours. Neutralisation and distillation are then proceeded with in the ordinary manner. The method is applicable to the determination of nitrates in drinking water, provided nitrites and ammonia be absent.

B. B.

The Presence of Ammonia in Zinc Dust. F. Robineau and G. Rollin (*Monit. Scient.* 1893, vii., 138, *through Chem. Zeit.*)—Commercial zinc dust appears always to contain ammonia, a fact the importance of which is obvious when it is used for reducing nitrates (see preceding abstract). On extracting zinc dust with warm water only a trace of ammonia is removed, as can be recognized by the use of Nessler solution, but on boiling the extracted material with caustic potash a further portion of ammonia is given off. Granulated zinc that has become partially oxidised by exposure to moist air, also contains ammonia. The complete removal of ammonia is conveniently effected by washing the zinc dust with boiling 1 per cent. sulphuric acid, after which treatment no ammonia is given off on heating with potash. As exposure to the air for two days

suffices to cause the reappearance of ammonia in purified zinc dust, it is necessary to prepare it as it is wanted, and use it immediately.

B. B.

Kreis' Modification of the Reichert-Meissl Process. (*Chem. Zeit.*, 1892, xvi. 1394; 1893, xvii. 395, 468.)—At the Annual Meeting of the Swiss Association of Analytical Chemists, held in September, 1892, Kreis announced that butter-fat and the fats used for margarine can be completely hydrolysed by strong sulphuric acid at 30-32°C. and that, after dilution, the volatile acids may be directly distilled over and estimated as usual. He noted that while the results obtained with pure butter by this method agree well with those given by the old saponification process, those obtained with margarine are much higher. This appeared to him to open the door to a new method, more delicate than we at present possess, for detecting margarine in butter.

Pinette (*Chem. Zeit.*, 1893, xvii. 395) has pointed out that the formation of sulphurous acid during the hydrolysis renders Kreis' modification quite impracticable as he stated it. By oxidising the sulphurous acid before distillation better results are obtained. Pinette proceeds as follows:—Five grams of the filtered butter-fat are melted in a convenient flask, and 10 c.c. of strong sulphuric acid are added with continual agitation. The fat immediately dissolves with evolution of sulphurous acid; 150 c.c. of water are added, and a strong solution of potassium permanganate until the pink colour is permanent for several seconds; the liquid may then be distilled. The rapidity of the method is greater than that of the Reichert-Meissl process, and Pinette is also of opinion that a comparison of the two processes will form a useful indication of margarine.

Prager and Stern (*Chem. Zeit.*, 1893, xvii. 468) propose to eliminate the sulphurous acid by a stream of air, succeeded by one of carbonic acid, and proceed as follows:—5 grams of the butter-fat are brought into a litre flask, 10 c.c. of strong sulphuric acid are added, and the flask is kept for ten minutes at 30-32°C. with constant agitation. When the liquid is cold, air is bubbled through it until the odour of sulphurous acid has disappeared, 100 c.c. of water are added, with precautions against rise of temperature, and carbon dioxide is bubbled through for ten minutes. This is then displaced by a stream of air for another ten minutes, the delivery tube is washed into the flask with 50 c.c. of water and the distillation is effected. The following results are quoted:—

C.C. of $\frac{N}{10}$ alkali required by five grams of butter-fat.			
		Reichert-Meissl.	Prager-Stern.
Sample a	...	29.86	29.60
„ b	...	30.23	29.65
„ c	...	28.34	27.76
„ d	...	28.20	28.10

The authors do not comment on the possibility of loss of acids other than sulphurous in the stream of air, but they admit that further investigation is requisite to render the Kreis suggestion serviceable.

A. G. B.

Determination of Fatty Oils in Mineral Oils. J. Klimont. (*Chem. Zeit.* 1893, xvii. 543.)—Fifteen grms. of the oil which is to be examined for the presence of small quantities of fatty oil are saponified in a flask, holding about 400 c.c., with 100 c.c. of an alcoholic solution of caustic potash (not soda), by boiling under a vertical condenser for one or two hours. The contents of the flask are then diluted with an equal bulk of water and filtered through a wet filter. The residue of mineral oil is well washed, the filtrate exactly neutralised with hydrochloric acid, allowed to stand in a separating funnel until thoroughly cold to get rid of any remaining mineral oil, and then extracted with petroleum ether to complete the removal of the unsaponified portion. The aqueous liquid containing all the fatty oil as soap, is concentrated to 100 c.c., precipitated with a solution of calcium chloride, and the resulting lime soap collected on a weighed filter, washed with the least possible quantity of cold water, dried and weighed. It is then ignited, and the residual lime weighed again, giving the fatty acids. These can be calculated to glycerides by adding the weight of the glyceryl radicle displaced by the lime, the necessary value being obtained by multiplying the weight of the CaO found by 0.774. The process is adapted for the determination of small percentages of fatty oils, e.g., under 5 per cent., but is not suitable for larger amounts, as the lime soap encloses a good deal of potassium chloride and calcium chloride, and cannot be satisfactorily washed.

B. B.

The Separation of Ferric Oxide and Alumina. H. Borntrager. (*Zeits. anal. Chem.*, 1893, xxxii., 187, through *Chem. Zeit.*)—The mixed oxides of iron and aluminium are ignited, weighed and dissolved in hydrochloric acid, neutralised with caustic potash (not soda), and precipitated hot with potassium oleate, avoiding an excess of the precipitant as it dissolves the iron and aluminium soaps that are thrown down. The precipitate is filtered, washed with hot water, and dried over calcium chloride to remove the bulk of the water which it contains. Hot petroleum is poured over the dried precipitate, and the ferric oleate is thus dissolved, leaving the aluminium oleate, which can be ignited, and the resulting alumina weighed. The solution of ferric oleate in petroleum is ignited in a platinum dish, and the residue of ferric oxide also weighed. The process can be used for the separation of ferrous iron from aluminium, as ferrous oleate is soluble in petroleum like the ferric salt.

[NOTE BY ABTRACTOR.—The usefulness of the method is impaired by the circumstance that alumina, if ignited sufficiently strongly to become anhydrous, is insoluble in hydrochloric acid, so that the mixed oxides would have to be dissolved by treatment with potassium bisulphate, or some equivalent method.]

B. B.

Detection of Monovalent Alcohols. B. V. Bitto. (*Chem. Zeit.* 1893, xvii. 611.)—The usual methods, such as that depending upon the iodoform reaction, for detecting alcohol, are not satisfactory, since they do not apply to alcohol only, but also to

other analogous substances. The reaction about to be described, although not particularly delicate, is claimed by the author to be definitely characteristic of monovalent alcohols. 0.5 grm. of methyl violet is dissolved in a litre of water, and 1 to 2 c.c. of this solution together with 0.5 to 1 c.c. of a solution of an alkaline polysulphide, is added to the liquid to be tested. If a monovalent alcohol be present, the liquid remains clear, but becomes cherry or violet red. It must be noted that a fair amount of the liquid to be tested should be taken, and that the coloration alters on standing. In the absence of a monovalent alcohol the liquid becomes greenish-blue, and after a time deposits reddish-violet flocks, the liquid itself, at the same time, becoming yellow. The reaction appears to take place with substances containing the group $C(OH)$. The behaviour of the following bodies has been tried:—

Substance.							Coloration.
Methyl alcohol	Cherry red.
Ethyl alcohol	"
Normal propyl alcohol	"
Iso-propyl alcohol	"
Tertiary butyl alcohol	Violet red.
Iso-butyl alcohol	"
Iso-butyl carbinol	"
Allyl alcohol	"

Divalent and trivalent alcohols, carbohydrates, acids, aromatic compounds, phenols, etc., do not give the reaction.

B. B.

The Analysis of Rubber Goods. D. Holde. (*Mitt. könig. tech. Versuchs.*, 1892, x., 315, through *Chem. Zeit.*)—The author supplements the work of Henriques (*THE ANALYST*, xviii., 13), by the suggestion of a method for the determination of the oil in rubber, and its surrogates other than that which has been "vulcanised" by treatment with sulphur chloride. The unaltered oil is soluble in ether-alcohol, whereas rubber and its surrogates are not much attacked by this solvent. It is true that pure caoutchouc yields three to four per cent. of an oily substance when extracted with ether-alcohol, and a correction is therefore necessary. The mixture adopted consists of four parts of ether and three of alcohol, and is best used by allowing the rubber to stand in the ether for a day, and become swollen to a pulp, the mass being heated, if necessary, and then adding the alcohol. After the removal of the fatty oil, the analysis can be proceeded with in the manner indicated by Henriques (*loc. cit.*)

B. B.

Coffee Glazing. F. Filsinger. (*Chem. Zeit.*, 1893, xvii., 498.)—In order to give a lustrous aspect to roasted coffee, a liquid is now in use which is free from colour, taste, and smell, and is clear and oily in appearance. It has a specific gravity of 0.868 at 15° C, and burns with a sooty flame, leaving no fixed residue. It absorbs no iodine when

THE ANALYST.

treated by Hübl's method, and is but little affected by chromic acid and concentrated sulphuric acid, taking only a slight brown colour when treated with the latter reagent. It mixes in all proportions with petroleum ether. It therefore appears to be nothing but a highly purified petroleum oil, and must be considered as an illegitimate addition.

B. B.

The Volumetric Estimation of Acetone. F. Robineau and G. Rollin. (*Monit. Scient.*, vii., 272, through *Chem. Zeit.*)—The usual method for the determination of acetone rests upon the conversion of that body into iodoform, which is weighed. The method is slow and inexact, owing to the loss of iodoform by volatilisation. These inconveniences are remedied by the author, who converts the method into one in which the estimation is volumetric. The acetone, having been obtained in a solution free from substances that also give the iodoform reaction, is treated with excess of potassium iodide and caustic soda, and a standard solution of sodium hypochlorite run in until the conversion of the acetone into iodoform is complete, as evidenced by the appearance of a blue colouration when a drop of the liquid, being titrated, is placed upon a piece of starch paper, moistened with a solution of starch and sodium bicarbonate. A mere trace of sodium hypiodite, in the presence of caustic soda, gives a blue colour with starch paper that is saturated with sodium bicarbonate. Certain precautions are necessary to obtain exact results. The liquid containing the acetone must be thoroughly alkaline with caustic soda, as otherwise more hypochlorite than the normal amount will be necessary to convert the acetone into iodoform. An excess of potassium iodide must be present. The dilution of the solution titrated must be nearly the same in all cases, and a similar strength of hypochlorite must always be used. The operation should not be conducted in a strong light, and the solution must be constantly stirred during titration. The standardisation of the hypochlorite is effected by means of pure acetone.

B. B.

Rapid Process for determining Organic Nitrogen in Urine. A. Petit and L. Monfet. (*J. Pharm. Chem.*, 1893, xxvii. 297, through *Chem. Zeit.*)—The process rests on the same principle of that of Kjeldahl, but with the modification that the ammoniacal nitrogen is finally estimated by means of an alkaline hypobromite. 10 c.c. of urine are digested with 5 c.c. of sulphuric acid in the usual way, with the addition of mercury, after which the solution is diluted with 20 c.c. of water and caustic soda added in small quantities at a time, the flask being kept cool with water. The liquid is left slightly acid, poured into a 50 c.c. flask, made up to the mark, and 10 c.c., corresponding to 2 c.c. of the urine, introduced into a urinometer and mixed with a solution of sodium hypobromite, made by adding 10 c.c. of bromine to 90 c.c. of strong soda lye and diluting with 75 c.c. of water. In the case of urine containing albumen the digestion with sulphuric acid is somewhat longer, taking about half-an-hour. For alkaloids containing a pyridine or quinoline nucleus, a digestion of $1\frac{1}{2}$ to 2 hours is requisite. For pyridine itself or analgesin even 4 hours is not sufficient to ensure complete oxidation.

B. B.

The Oils from the Kernels of Apricots, Cherries, Plums and Peaches, and their possible use as Adulterants of Almond Oil. C. Micko. (*Zeits. österr. Apoth. Verein.*, 1893, xxxi. 175.)—The above-named oils are all fluid at the ordinary temperature and have a yellow colour and a mild pleasant taste recalling that of almond oil, so that they could not be detected in admixture with it by simple inspection. The author has therefore determined the following constants by means of which discrimination is practicable:—

		Apricot.	Cherry.	Plum.	Peach.
For the oils	Specific gravity ...	0.92108	0.92850	0.91949	0.92147
	Acid number ...	0.64	1.11	0.55	0.52
	Saponification ...	193.11	193.36	191.55	191.11
	Iodine number...	108.07	114.30	100.20	99.71
For the insoluble fatty acids ...	Saponification number...	194.04	189.04	200.47	200.87
	Iodine number ...	102.64	104.33	104.21	101.93
	Melting point...	13.4—18°C	16—20.6	*12.4—18.1	10—18.9
	Mean molecular weight	288.60	296.23	279.34	278.78

The iodine absorption is therefore the best single criterion for these oils. Certain colour reactions are also given as an aid in detecting them. With nitric acid of specific gravity 1.4, apricot and plum oil give an orange colour, peach oil at first a yellowish brown then a dirty orange; cherry oil gives a dark brownish red colour and the aqueous layer becomes bright red on standing. Bieber's reagent (a mixture of concentrated sulphuric acid, crude nitric acid and water in equal parts), gives a beautiful peach-red colour with apricot oil, a brown, darkening on standing, with cherry oil, a rose colour with plum oil, and with peach oil no colouration for the first half-hour, but a bright brown colour after the lapse of some hours. It has hitherto been generally accepted that peach oil gives a peach-red colour, but according to the author's experiments this is not the case, that colour being characteristic for apricot oil.

B. B.

Phosphates in Milk. Duclaux. (*Ann. Inst. Past.*, 1893, vii. 2, through *Chem. Zeit.*)—According to the experiments of the author, the phosphates in milk are present in suspension and solution. The former are phosphates of iron, aluminium, magnesium and calcium, while the latter consist of nearly equal molecular quantities of phosphate of sodium and phosphate of calcium, held up by sodium citrate. The result of the examination of various kinds of milk is that there is about twice as much calcium phosphate in suspension as in solution, and that the composition of the ash of milk of the most diverse origins is very nearly identical. An attempt to produce a "phosphate milk" by adding phosphates to the fodder has proved quite ineffective, the milk containing no more phosphate than ordinary qualities.

B. B.

The Analysis of Aniline Oil. H. Reinhardt. (*Chem. Zeit.*, 1893, xvii. 413, 414.)

—The method used by the author depends upon the following facts:—

(1.) Aniline, when treated with a mixture of potassium bromide and bromate in dilute acid solution, yields the tribromo derivative, while ortho- and para-toluidine give dibromo substitution products.

(2.) On precipitating a hydrochloric solution of the three bases with oxalic acid, under suitable conditions, para-toluidine is first thrown down, then aniline, while ortho-toluidine remains in solution. The author's control analyses have been carried out with the pure bases prepared by himself, as the commercial products are not sufficiently good for the purpose.

I. The determination of aniline and of the total amount of the two toluidines in mixtures of aniline and either or both toluidines.

The brominating solution is prepared from a solution of 480 grms. of bromine, 336 grms. of caustic potash of 100 per cent. strength, dissolved in 1 litre of water, the whole being boiled for two or three hours in order to get rid of hypobromite, and finally diluted to 9 litres. For the standardisation of the solution as well as for the actual determination of the oil, about 1.5 to 2 grms. of the oil are taken, dissolved in 100 c.c. of hydrobromic acid of specific gravity 1.45 to 1.48, diluted with a litre of distilled water and titrated with the bromine solution until there is present sufficient excess of bromine to give a colouration with paper that has been soaked in a mixture of potassium iodide and starch. The bromine solution retains its strength fairly well, a very trifling difference being perceptible after it had been kept a fortnight.

That the method is reasonably exact is shown by the fact that the results for pure oils ranged from 99.90 to 100.06 per cent. The content of aniline is reckoned by the following equation:—

$$x = 2.3777vt - 1.3777a$$

where a is the quantity of oil taken, x the amount of aniline it contains (that is to be determined), v the volume of bromine solution used in c.c., and t the amount of aniline to which one c.c. of the bromine solution corresponds as determined in the process of standardisation. The weight of aniline found in this manner, deducted from the total quantity of oil taken for analysis, obviously gives the amount of the two toluidines, and the percentages can then be calculated in the ordinary way. The result of test analyses are very satisfactory.

II. The determination of para-toluidine in the presence of aniline or ortho-toluidine or of both bases.

THE ANALYST.

In order to obtain accurate results by the oxalate method mentioned above, more oxalic acid must be used than suffices for the precipitation of the para-toluidine present. A preliminary trial must therefore be made to gather some idea of the content of para-toluidine. The quantity of oil taken for analysis varies from 10 to 100 grams, according to its content of the body to be estimated. In general, 100 grams of the oil are mixed with 106 grams of hydrochloric acid, free from sulphuric acid, and containing about 31 per cent. of HCl, and the mixture added to a solution of the amount of oxalic acid (free from lime) that has been found necessary by the preliminary trial, dissolved in ten times its weight of water. The resulting liquid should remain clear even when a large percentage of para-toluidine is present; crystallisation of the oxalate takes place after stirring and is completed by allowing the solution to stand for 48 hours. The oxalate is then freed from the mother liquor by filtration with the pump, and washed three times with distilled water, 25 c.c. being used for each washing. The oxalate is then decomposed by being added to a solution of caustic potash (190 c.c. of potash, 45°B, and 200 c.c. of water), and after cooling, the precipitated oil collected and weighed. It contains a certain amount of aniline, which is determined by titrating it with bromine solution in the manner already described. A simple calculation gives the percentage of para-toluidine, to which a constant correction of + 2 per cent. must be made for the prescribed conditions of separation. The test analyses given are very satisfactory. The use of these methods of analysis shows that the commercial products purporting to be pure oils, often contain considerable quantities of their isomers or homologues.

B. B.

On Electrolytic Methods of Analysis. F. Oettel. (*Chem. Zeit.*, 1893, xvii, 173 174.)—The author performs the very necessary task of calling attention to the want of precision in the methods commonly adopted for describing the conditions under which electrolytic processes should be carried out. It is the custom simply to state that with a current of so many amperes the metal under consideration separates out in a state of purity, and in a compact and weighable form. This is quite insufficient to enable another analyst to repeat the operation with the certainty of success, as the most important factor in the separation of a metal in a given condition, is not the strength of current, but the current density, that is to say the number of amperes per unit of area of the electrode. He therefore proposes that all who record the results of investigations of this kind should select the square decimeter as the unit of area, and state that the separation was effected by a current of so many amperes per square decimeter of cathode. It is important to note that the current density will not be uniform over the surface of the cathode if the latter be in the shape of a cylinder, for example, surrounding a wire which serves as the anode. In this case the density will be greater on the inner surface, and more metal will therefore be deposited there. It suffices in such cases to state the average current strength referred to the whole immersed surface of the cathode, and to mention the shape and disposition of

the cathode, the object being not the attainment of rigid accuracy in stating the current density, but the indication of it in such manner as will enable another chemist to reproduce the conditions which experience has shown to be successful. Another objectionable practice in recording the results of electrolytical researches, is that of stating the current in c.cs. of explosive gas given off in a voltmeter in circuit. The strength should always be given in amperes, about which there is no ambiguity. It may not be needless to add, in view of the mistakes that are often made, that the measuring instrument, whatever it be, must be inserted in circuit while the analytical operation is in progress, as a wholly false result will be got by substituting it for the depositing cell when the latter is removed. A convenient form of amperemeter is a cell containing two copper plates immersed in a solution of 15 parts of copper sulphate, 5 of sulphuric acid, and 100 of water. The gain of weight of the cathode is the measure of the current that has been employed, and is reduced to amperes by the use of the equation,

$$a = \frac{w}{t \times 0.0197}$$

where a represents the current in amperes, t the time in minutes, and w the weight of copper in grams. The advantage of the copper voltmeter is that as there is no reduction of metal, but only its transference from anode to cathode, the instrument consumes only the energy required to overcome its internal resistance, which may be small, and thus the drop of voltage which it occasions is in no way considerable.

The author also calls attention to the desirability of stating the voltage of the current used, although this is of less importance than the current density. B. B.

[NOTE BY ABTRACTOR. It has been recently shown that the electrolytic separation of metals, the heat of combination of whose salts differs appreciably, can be conveniently effected by the use of a current of regulated pressure, which may be arranged to throw down those whose heat of combination is lowest, while leaving the others in solution. The voltage of the current employed, therefore, cannot be neglected.]

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Summation 50

Thompson 15

GLAMORGANSHIRE QUARTER SESSIONS.

THE SALE OF BAKING POWDER CONTAINING ALUM.

[Compiled from Reports in *The Cambrian* and elsewhere.)

WEDNESDAY, APRIL THE 12TH, 1893.

ON the bench:—J. Coke Fowler, Esq., Chairman; his Honour Judge Gwilym Williams, Vice-Chairman; and Edward Daniel, Esq.

This was an appeal case brought by James James, grocer, Blaenrhondda, who was convicted at Pontypridd on February 13th last, under the Sale of Food and Drugs Act, for selling baking powder, known as the "Excelsior" baking powder, containing injurious ingredients. Mr. D. Brynmor Jones, Q.C., M.P., Mr. T. C. Blofield (specially retained) and Mr. Arthur Lewis (instructed by Messrs. Tillett and Co., Norwich), appeared for the appellant, and the respondent

(Supt. Evans, Pontypridd) was represented by Mr. David Lewis and Mr. Rhys Williams (instructed by Mr. W. E. R. Allen, Cardiff).

In opening the case for the respondent, Mr. David Lewis said he thought there would be no dispute as to the ingredients of the baking powder in question. There was no definition as to what baking powder really was, but when not adulterated with alum or anything else, it was entirely bi-carbonate of soda. The question of the constitution of Borwick's baking powder had often been brought up. That was made in a certain way, not of alum but of tartaric acid. When he told them that tartaric acid cost over £6 per hundredweight, whilst alum was only five shillings, they would readily see that the temptation to use alum was very great indeed. Therefore, it was desirable that the public should be protected in this matter. The learned counsel then explained the chemical changes which took place when alum came in contact with the saliva and gastric juice of the mouth and body of a person, with a view of showing that the chloride of aluminium which was formed was injurious to the system, by reason of its retarding the digestive operations.

Superintendent Evans was then called and examined by Mr. Rhys Williams. He said he was stationed at Pontypridd, and was an inspector appointed under the Food and Drugs Act. He then went on to depose to the purchase of the baking powder from the appellant on December 10th last. According to Dr. Morgan's (public analyst) certificate the substance contained alum to the extent of 30 per cent., which was, in Dr. Morgan's opinion, prejudicial to health. Witness went on to speak of subsequently making bread with the powder in it, and submitting the loaves to various medical gentlemen for analyses.

Dr. W. Morgan (Public Analyst) was next called, and stated that taking a teaspoonful of the baking powder at 120 grains, a four-pound loaf would contain 360 grains, four-tenths of which would be alum. When water was added to the baking powder, chemical action took place between the potash alum and bi-carbonate of soda, and produced hydrate of alumina, sulphate of soda, sulphate of potash, carbonic acid gas and water. The quantity of hydrate of alumina might be taken as one-sixth of the alum, or six grains per pound loaf. When the bread was eaten the hydrate of alumina was dissolved by the gastric juice in the stomach, in consequence of the hydrochloric acid and pepsin contained in the gastric juice. The result was that chloride of aluminium was formed from the contact between the hydrate of alumina and free hydrochloric acid, and that chloride of aluminium was noxious to the stomach. Detailing a personal experiment made a few days ago, Dr. Morgan said that he and his son partook of an ordinary mid-day meal. His son drank some hydrate of alumina in water with his food, but he, himself, drank nothing. The hydrate of alumina was obtained from the baking powder in question. A short time afterwards artificial vomiting was produced, and hydrate of alumina was then added to the contents of his own stomach. Both vomits were then dialysed and chloride of aluminium was found in each, thus proving that the hydrate of alumina was in a soluble state. That experiment had since been twice repeated, and on the first occasion was made under the supervision of Drs. Griffiths and Brooks. Cross-examined by Mr. Brynmor Jones, Dr. Morgan admitted that though an oxide might be injurious, a compound might be harmless, as was shown in the case of common salt, which was purely chloride of sodium, yet chlorine was poisonous. In the making of bread, however, he was of opinion that alum would produce the same effect as alumina.

Mr. Otto Hehner, called, said he was past President of the Society of Public Analysts and Public Analyst to several counties in England, and had made food analysis his special study for the past twenty years. In his opinion such baking powder as that in question was injurious to health by reason of the aluminium salts it contained. He was of opinion that hydrate of alumina was soluble in the stomach. Cross-examined by Mr. Brynmor Jones: Witness had written a good deal upon food analysis, and he, as well as many medical men, considered alum an abomination in bread. Alum and alumina were two distinct substances producing the same result.

Professor Claude Thompson, Professor of Chemistry in University College, Cardiff, gave substantially the same evidence, and detailed experiments he had made, coming to the conclusion that hydrate of alumina was soluble in the stomach. Cross-examined by Mr. Brynmor Jones: Witness said that when alum was dissolved in water there was no chemical change. He could not say what would be the result if the alum when mixed with water were heated.

Professor Wyndham Dunstan, M.A., Oxford, F.I.C., Professor of Chemistry at St. Thomas's Hospital, London, &c., was next called, and he stated that the hydrate of alumina, dried at 212° F., was insoluble in water, but was soluble in the gastric juice of a dog diluted to 0.2 per cent. strength, and that the gastric juice of a dog diluted to this extent dissolved hydrate of alumina from bread baked with the powder. That the hydrate of alumina in the bread

interfered with the digestion of starch by ptyalin (diastase was used in the actual experiment) and with both peptic and pancreatic digestion. Further, that dilute solution of carbonate of soda of 0.3 per cent. strength, the strength of the alkali in intestinal juice, would dissolve hydrate and phosphate of alumina, either freshly precipitated or dried at 212° F., so that absorption of aluminium compounds could occur from the intestine. This was confirmed by his observation that aluminium compounds occurred in the urine of a man who had daily taken 15 to 30 grains of the hydrate dried at 212° F. On one occasion the taking of this quantity of hydrate produced vomiting. In the course of a subsequent re-examination, Professor Dunstan said he had heard Dr. Morgan relate his experiments, which in his (witness's) opinion made the matter more conclusive, and were very valuable experiments.

Dr. Lauder Brunton was next examined, and stated that it was a fact that the action of alum caused disturbance of the digestive functions. He was of opinion that Dr. Morgan's experiments afforded conclusive evidence that the hydrate of alumina in the stomach changed into chloride of aluminium, the cause of the change being the hydrochloric acid. The presence of alumina in the stomach he considered injurious to the human system. In cross-examination by Mr. Brynmor Jones, the witness stated that alum taken in small quantities had an astringent action, but when a larger quantity was taken, it had an irritant action. It was the constant use of baking powder containing alum that proved so injurious to a person. Hydrate of alumina had the same effect as alum, because the base was the same in one respect.

The Court then adjourned till Thursday.

THURSDAY.

Dr. Rhymer Marshall, Doctor of Science of the University of Edinburgh, and Fellow of the Chemical Society, but now of University College, Cardiff, was the first witness called and examined by Mr. David Lewis. He agreed with the previous witnesses for the respondent that hydrate of alumina was passed into the stomach by the eating of alum, and that that hydrate was soluble in the gastric juices and formed chloride of aluminium. In Scotland bi-carbonate of soda was frequently used as a baking powder.

Dr. Thomas Druslyn Griffiths, of Swansea, stated that there were two effects—local and constitutional—produced when hydrate of alumina was converted into chloride of aluminium in the body, whilst the result was highly irritating to the internal organs. Cross-examined by Mr. Brynmor Jones: He had never treated a patient suffering from the effects of this irritant, and had declined to give evidence in the present case on either side until he saw Dr. Morgan's experiments.

Dr. Ebenezer Davies, Medical Officer of Health for Swansea, gave similar evidence as that previously given, submitting that six grains of hydrate of alumina retarded the progress of digestion.

Dr. Thomas Henry Morris, Bachelor of Medicine of the Glasgow University, now practising in the Rhondda Valley, stated that prior to this case he was not aware that alum was used in baking powder. Taking the proportion as used in the "Excelsior" baking powder, it would be highly injurious to health.

Dr. W. Williams, Master of Arts, and Bachelor of Medicine, of Oxford, &c., and Medical Officer of Health to the Glamorgan County Council, was the last witness examined on behalf of the respondent's case. He said that an antidote in a case of poisoning by alum must be an alkaline one, because the acid was the principal substance to be neutralised.

The Court then adjourned for luncheon.

Directly after luncheon, Mr. Brynmor Jones opened the case for the appellant, his address lasting nearly two hours. At the outset he read section 3 of the Act under which the proceedings were instituted, and said the only evidence given by his learned friend of an offence under the Act, was that James James, the appellant, sold a packet of baking powder. The case was, therefore, not the same as if the appellant had sold a loaf of bread which had been manufactured with the aid of the powder. Now was baking powder food? He would submit that it was not used for the food of man.

The Chairman: You must take it in connection with the interpretation clause of the Act.

Mr. Jones replied that he did so, and went on to say that what was meant by food in the Act was substances taken for the purpose of sustaining the life of human beings. The mere fact that some substance used for the making of food ready for reception into the stomach of a person did not render such articles, mixed in the preliminary stage of the manufacture of food, food itself.

The Chairman said he felt the force of the argument and could not see how the Bench could possibly hold that baking powder in itself was food. But the interpretation clause certainly seemed to suggest a latitude. The Act of Parliament was a remedial one passed *pro bono publico*, and that being the case there ought to be a liberal interpretation of the section.

Mr. Jones opposed to that that the Act of Parliament was not to be construed extensively, and went on to show that it was a penal statute. But if the learned Chairman's construction was accepted, that baking powder was an article of food, what, the learned counsel asked had been "mixed" with it, in order to constitute an offence under the section? The evidence for the prosecution did not show that anything had been mixed with the powder.

The Vice-Chairman: Baking powder need not necessarily contain alum.

Mr. Jones: It is absolutely necessary, according to the evidence already called, in order to make baking powder. Bi-carbonate of soda without an acid was not a baking powder at all.

The Vice-Chairman: But it is not a baking powder until allied with bread.

Mr. Jones: Then it is only an offence if we had sold a loaf.

The Vice-Chairman believed that baking powder, in the abstract, was an article of food, because it became a part of the loaf. The case for the prosecution was that some portion of the baking powder remained as a residuum, and that it became a part of the bread which went into the stomach. If so, surely the powder was food. If the learned counsel could persuade him that in the process of kneading the dough or baking it, the whole of the alum, which was admitted to be in the powder, was disposed of, then there would be no case.

Mr. Jones: The evidence shows that the whole of the alum in the baking powder is necessarily got rid of, and a new substance created.

The Vice-Chairman: No, it is alum in another form, and would not be there unless you put the alum in the flour.

Mr. Jones differed, and went on to refer to the composition of water with a view of showing that the hydrate of alumina was no more a form of alum than water was a form of either oxygen or hydrogen, after which he read a decision of the Recorder of Cambridge (Mr. Bullwer, Q.C.), that baking powder was not an article of food. He further submitted that there was no evidence adduced to show that the baking powder was injurious to health, but only that it might be. According to the form of the indictments and summonses which were usual in their criminal practice, the offence must always be charged with certainty.

Mr. Blofield concurred with all that Mr. Brynmor Jones had said, but the objections were overruled, the Bench considering there was a case to answer.

Mr. Brynmor Jones then continued his address, and said the use of baking powders had gone on for a period of fifty years, as it was well known that unleavened bread was very indigestible. He explained that the object of baking powders was to liberate carbonic acid gas and so cause the dough to rise. One of the earlier substances used as a baking powder for that purpose was made from carbonate of soda and tartaric acid, whilst a later method was the introduction into dough of carbonic acid gas by mechanical appliances, as was the case with the aerated bread. But alum baking powders had been known for many years, and alum was quite as essential a substance to use as tartaric acid.

The Chairman: But you would not say that alum tends to increase fermentation?

Mr. Jones: Yes; only indirectly it produces carbonic acid gas. Learned counsel then went on to say that Dr. Dyke, one of the witnesses whom he should call, had used an almost identical composition as the present baking powder in his family for a great many years. That powder Dr. Dyke prepared himself, and he would say that neither himself nor his family ever felt any injurious effect from its use. Further than that, he would call evidence to show that of the immense amount of "Excelsior" baking powder, which was manufactured at Doncaster, and also sent out from Norwich, not a single patient coming under the care of the professional witnesses that would be called, had ever suffered from dyspepsia or other ailments from its use. The only difference between the alum baking powders and that made from tartaric acid was that the former had a slower action, and many persons consequently preferred it. Having referred to the process of digestion of food in the body, remarking that it was the ptyalin of the saliva that was the principal agent in this respect, the learned counsel observed that it was a remarkable fact that though the Cambridge decision was given eleven years ago, yet there had only been one similar prosecution, that at Ilston, where there was also no conviction, since that time, and assigned as the cause that nobody thought fit to question that decision. The reason why he had been so particular as to mixture and chemical combination was because alum was formerly used in bread, not for the purposes of a baking powder, but in order to make inferior flour look white, and it was a vulgar belief that because an element was harmful, a chemical combination was also harmful. Inspector Jones had been labouring under that vulgar belief.

and he was sorry to say the public analyst (Dr. Morgan) had also confirmed that when he said on the certificate, "In my opinion the use of alum in bread is prejudicial to health." It was the metal aluminium—a double salt—in combination with water that was formed, but that was a totally different thing, and, as he would show, was injurious. Learned counsel also denied that hydrate of alumina was soluble in the stomach, and said that he would call in support of his contentions Dr. W. Richardson, who was known throughout the world; Dr. Luff, Mr. Wynter Blyth, Dr. Dyke, Mr. Sutton, and many other eminent medical men, many of whom had written largely upon questions affecting their profession. In conclusion, he said he could say without hesitation that the theory he should place in opposition to the theory of the prosecution would be quite as strongly supported, and if the learned Bench did not believe the case for the appellant exactly preponderated, yet they must give him the benefit of the doubt.

Mr. Francis Sutton, Fellow of the Institute of Chemistry and Public Analyst to the County of Norfolk and the town of Great Yarmouth, was then examined by Mr. Blofield. He and his family, he said, had used the "Excelsior" baking powder for upwards of thirty years without any ill-effects. He did not believe hydrate of alumina was soluble in the gastric juice, but passed through the intestines undissolved. With regard to Dr. Morgan's experiments, he (witness) did not consider them fair, as he took freshly precipitated hydrate of alumina, which was much more soluble than when it was baked in bread.

Replying to the Vice-Chairman, Dr. Morgan said he did not actually mix the hydrate with the bread, but wrapped it in a muslin bag and introduced it into the dough, after which it was baked as near as possible at the same temperature as other bread would have been.

The Court then adjourned till Friday.

FRIDAY.

The cross-examination of Mr. Francis Sutton, County Analyst of Norfolk and Borough Analyst of Great Yarmouth, was continued by Mr. David Lewis. On Thursday, it was stated by witness that an experiment had been performed upon a groom of Dr. Latimer. It was as follows:—The coachman was given a pound of bread made with the baking powder in question, with the proportions of bread to flour given upon the wrappers of each packet. He commenced to eat it at 6.50, and finished at about 7.15, and to assist him he took three glasses of water, or a pint and a half in all. He then went out for a walk and returned about 9.30 o'clock, when, in the presence of Mr. Sutton, Dr. Luff, and Mr. Wynter Blyth and others, the tube was put in his stomach with a view of getting its contents up through it. Before, however, this could be done, he vomited about a pint of the contents of his stomach. This quantity was put into a dialyser, and left for the night in a room, which was locked, the key being taken by Mr. Sutton. On the following morning the dialysate was examined, and Mr. Sutton alleged that it did not contain chloride of aluminium, as it should have done if the theory of the prosecution was true. Mr. Sutton now maintained that this experiment was a conclusive answer to the experiment by Dr. Morgan upon himself and his son, and further stated that it was carried on under precisely the same conditions. However, upon being further asked by Mr. Lewis, witness stated that the dialyser used in his case was the bladder of a freshly killed sheep, and not, as was the case in Dr. Morgan's experiment, one made of vegetable parchment. Witness also admitted that if there had been chloride of aluminium in the contents of the stomach, it would probably have combined with the albuminoids present in the bladder; whereas in vegetable parchment there would be nothing to combine with chloride. Replying to further questions, he said he was of opinion that hydrate of alumina was insoluble when eaten with bread. About 20 years ago he was consulted by a manufacturer of an alum baking powder as to whether it was injurious. That powder contained about 35 per cent. alum, and after adopting its use in his family and upon himself, as well as upon a dog, he gave a testimonial to the effect that it was harmless. He knew that Borwick's baking powder had obtained many medals at exhibitions, and that the "Excelsior" had not any, but he had no interest in either. Witness also stated, with regard to the albumen contained in the sheep's bladder combining with the chloride of aluminium, he and Dr. Luff and Mr. Blyth had that morning tested the bladder itself, and found there was no chloride present. He admitted that at home he himself generally used a vegetable parchment dialyser, but thought a bladder was fit for the purpose, and, in answer to the Bench, said he was satisfied with the experiment until Mr. Lewis had, in cross-examination, pointed out the difference. Being cross-examined as to whether carbonate of soda by itself would serve the purpose of baking powder by giving off much carbonic acid gas under heat when mixed in flour, he would not

admit that that was so, but said that if it were added to flour and the whole mixed with sour milk, carbonic acid gas would be given off, and it would then act as baking powder.

Dr. Arthur Pearson Luff, Bachelor of Science of the University of London, Member of the Royal College of Physicians, Fellow of the Institute of Chemistry, Physician and Lecturer at St. Mary's Hospital, London, Official Analyst to the Home Office, &c., was next called. He stated that alum and alumina were absolutely dissimilar substances in this way; alum was a soluble body, hydrate of alumina was insoluble; alum was astringent, hydrate of alumina was quite non-astringent; alum contained aluminium as a sulphate, while hydrate of alumina did not, but was an altogether different substance. As a specialist on poisons, he might say that alum by itself and in certain quantities was a somewhat injurious substance, but hydrate of alumina was an inert substance and entirely harmless. He was also of opinion that this substance was absolutely insoluble in the acids of the gastric juice.

The Vice-Chairman: Can you conceive gastric juice operating upon bread containing this baking powder in such a manner as to render the hydrate of alumina soluble?

Dr. Luff replied that he could, in the case of the gastric juice of a dog, because of its strength, but not in the case of a human being.

The Chairman: What is the inducement to use alum baking powders?

Dr. Luff: I should say there are two reasons, one is that alum is so much slower in its generating action than tartaric acid, which means a good deal to a poor woman who has not always got her oven ready. The second reason is that, weight for weight, the alum baking powder is a cheaper powder.

In adversely criticising Dr. Morgan's experiment, the witness first paid a high tribute of praise to the Swansea Public Analyst as a skilful practitioner.

The Chairman: I am glad to hear you make that preface, as we also have a very high opinion of Dr. Morgan.

Dr. Luff, continuing, said he regarded the experiment as utterly valueless, as Dr. Morgan did not use hydrate of alumina as it existed in cakes or bread made with the alum contained in the baking powder in question. He simply took freshly precipitated hydrate of alumina which was not diffused in the flour and baked in the bread, and that made all the difference. With regard to inserting the substance in a muslin bag and then baking it in the centre of a loaf, that also rendered the experiment utterly valueless, inasmuch as the temperature of the interior of the loaf was not the same as on the outside for instance. It was put in the coolest part of the loaf, and consequently, not being distributed throughout, it was not a comparative experiment that could be taken the same as hydrate of alumina baked in bread. Further, Dr. Morgan gave from 15 to 30 grains of hydrate of alumina, and that would represent a quantity, in baking powder, which would be contained in about six pounds of bread. Nobody could eat that amount at once, and therefore the experiment was not a fair nor a comparative one. Would it be fair, he would ask, for him to give a man from 15 to 30 grains of arsenic, and assume that the action would be the same as though a smaller dose were given? They often gave small doses of arsenic, but they all knew that if a larger quantity were taken it was very poisonous. The witness then detailed experiments he had made with a view to ascertaining whether hydrate of alumina was soluble or not. He first prepared hydrate of alumina, and then digested it in test tubes with a solution of .02 per cent. of hydrochloric acid, as representing, as near as possible, the percentage of acid contained in the human gastric juice. In none of his experiments had chloride of aluminium been formed, but, on the contrary, the hydrate of alumina was insoluble. Witness next repeated Mr. Sutton's evidence as to an experiment that had been tried upon Dr. Latimer's coachman in the presence of the witness and the other leading witnesses for the appellant. In this the coachman ate a pound of bread made with the baking powder in question. The contents of the stomach were afterwards digested and dialysed in the same way as Dr. Morgan's, but no chloride of aluminium was found.

Witness also said that his opinion that hydrate of alumina, baked in bread according to the directions on the wrapper of these powders, was insoluble in the gastric juice of the human body, was based upon the experiment made by Mr. Sutton, already detailed by him, upon the experiment made by himself about three weeks ago, and lastly, the experiment upon Dr. Latimer's coachman. The experiment made by himself three weeks ago was as follows:—He took some alum and powdered it in a mortar, and then added some hot distilled water, and rubbed them together until the alum dissolved. He filtered the solution through the best Swedish filtering paper, and warmed the mixture up to a temperature which he could only give approximately—from 150 to 200 degrees Fah. He did not boil the mixture. He then precipitated the hydrate of alumina and collected it on a Swedish filter, and washed it with distilled water heated to 212° Fah. He washed it for three hours, until there was nothing soluble

left. He ascertained that all the soluble substances were washed away. He next removed the hydrate of alumina from the filter-paper, and placed it in a platinum dish, and then put it in a drying oven at a temperature of 150° Centigrade. The bath used was entirely of copper. He baked the hydrate at 150° Centigrade, i.e., 302° Fah. for three-quarters of an hour. He then took different quantities of this hydrate of alumina and digested them in test tubes. He did not analyse the compound before doing so. He knew the compound as dried hydrate of alumina. He could not say what its formula was. He thought it was $\text{Al}_2(\text{HO})_6$; or the same minus a little of the water. A little of the water in combination might have been removed. It might have been oxy-hydrate, which was a different body from hydrate of alumina. He digested this in different test tubes, putting in each tube about five grains, with diluted hydrochloric acid of the strength of '02 per cent., which he believed to exist in gastric juice, at the temperature of the human body in health, viz., 98.5° Fah. There were between 35 and 40 cubic centimetres. The different tubes were digested for different periods, for one hour up to eight hours. He had a dozen tubes and tested the contents of eight. In the first of these digesting had gone on for an hour, in the second for two hours, in the third for three, and so on, each for a hour longer than the others up to the eighth, in which digesting went on for eight hours. At the end of each hour he removed one tube, filtered its contents, and tested the filtrate by the addition of a solution of ammonia, to see if any alumina or hydrate of alumina had been dissolved. No alumina compound had been dissolved. He tested in cold and in heat in every case, and used both tests—concentration and the other. The experiment was done on two days—Tuesday and Wednesday—in the same week. He believed that a '02 per cent. hydrochloric acid was the strength of this acid in human gastric juice. He relied upon Halliburton, the most recent authority, for this, and upon nothing else.

In further cross-examination, he admitted that the only authority for this weak acidity was a table quoted by Halliburton from experiments by Schmidt in 1855, and that Halliburton, while quoting the results, actually pointed out that the acidity was below the normal, presumably owing to the fact that the juice had been obtained from a person suffering from gastric fistula. Also that Halliburton, like other physiologists, accepted 0.2 per cent. as the strength to be used in making artificial gastric juice.

Witness was further cross-examined as to the kind of dialyser that was used, but he was of opinion that the sheep's bladder which they used was equal to the parchment used by Dr. Morgan.

Re-examined by Mr. Brynmor Jones: When alum was introduced into dough according to the directions given on the packets of "Excelsior" baking powder, it was altered by the heat when baked, and no alum was left at all, only hydrate of alumina, which was inert and insoluble in the stomach, and therefore not injurious.

Dr. Benjamin Ward Richardson was next called. Mr. Arthur Lewis was about to read out a lengthy list of the witness's qualifications, when the Vice-Chairman asked if it was necessary. Wouldn't it be sufficient to say the "great Dr. Richardson?" (Laughter.)

Mr. Arthur Lewis (to the witness): You are the great Dr. Richardson?

Dr. Richardson, laughingly: I don't say that.

Replying to questions put him by the learned counsel, Dr. Richardson said he had been 43 years in practice as a physician in London, and amongst other things had specially concerned himself with food analyses, including bread and its manufacture. The reasons why alum was first used were to stop excessive fermentation, and also, probably, to whiten bread. It was common information that there was a great difference between alum and hydrate of alumina. He had had samples of the baking powder in question, and had adopted its use in the bread he had eaten for the past three weeks; but he had not suffered the slightest inconvenience from its use, nor found any trace of aluminium in his constitution. He was of opinion that the use of the alum as contained in the "Excelsior" baking powder was not at all injurious to health.

By Mr. Arthur Lewis: You have heard it suggested in the course of the evidence for the respondent, that chloride of aluminium is formed in the stomach from the hydrate of alumina introduced there?

Dr. Richardson: I don't think that proved, but I would give way to experiment if satisfactory.

Mr. Arthur Lewis: Supposing that chloride of aluminium be formed in the way suggested, in your opinion would the chloride of aluminium so produced by the eating of bread made with this powder be injurious to health?

Dr. Richardson: I don't think it would get into the circulation at all if it were formed.

Mr. Arthur Lewis: And, therefore, would it be injurious to health or not?

Dr. Richardson: It would have nothing more than a local effect, experienced at the time the bread was digested.

Continuing, Dr. Richardson said he did not know of a single case where a patient suffered from any ailment from the use of alum baking powders.

By Mr. Arthur Lewis: What do you say, in regard to the suggestion of Dr. Lauder Brunton, that bulbar paralysis may result from the hydrate of alumina?

Dr. Richardson: I know nothing about it at all, and I rather regret that my learned friend should have made such a suggestion. It is a venture—a new suggestion—with no basis whatever.

Cross-examined by Mr. David Lewis: He had not had time to make any experiment. He heard Professor Dunstan say he had found chloride of aluminium. He would not say he disagreed with him. It was just possible that in some cases there might be a gastric juice of more than usual acidity, which might cause chloride of aluminium to be formed. It was his impression that it had been stated there by the other side that chloride of aluminium was absorbed into the system. He could not controvert that hydrate of alumina was soluble in sodium carbonate. The treatises mostly considered .2 per cent. as the strength of the gastric juice. As to .02 per cent., he was afraid to make any statement. As to .2 per cent., he would rather not answer. It would be brought against him again. He had made experiments, but they were not satisfactory. They wanted a series of experiments to prove that, as it was just possible that that particular gastric juice might have been more than usually acid, in which case there might have been a temporary formation of chloride of aluminium. He did not think that this substance could possibly be absorbed into the system in consequence of the coagulation of the blood. Since he had been in Swansea he had had a sheep killed for the purpose of verifying that point. He found that chloride of aluminium instantly coagulated the blood. He had made special enquiry into the question of baking powders containing alum, and he had come very clearly to the conclusion that from alumina hydrate there was sometimes caused constipation and indigestion. He admitted that, but it never went further than a mere local effect. It never had a constitutional effect. Before giving place to the next witness, Dr. Richardson asked to be allowed to make a statement on the matter.

The Chairman consented.

Dr. Richardson then went on to say that he had controverted some of the theoretical views of his colleagues, and would yield all to them, but he would say that where there was not a single instance of injuriousness, they could not fairly charge a man with a criminal offence who sells—

The Chairman: We cannot allow you to make any observation like that.

Dr. Richardson: Then I put that aside; but when a substance which is taken with impunity by hundreds and thousands of people, and yet has never resulted in an inquest—

The Chairman: I am afraid you are assuming the position of advocate. (Laughter.)

Dr. Richardson: And a substance that has never given a *post-mortem* examination, I say it is impossible that it can be injurious to the system, and it is like straining at a gnat and swallowing a camel to say so. (Laughter.)

Mr. David Lewis: It is not like alcohol. (Renewed laughter.)

Dr. Richardson, who is the well-known temperance physician, replied, amid laughter, "No, it is not."

John Hughes, coachman to Dr. Latimer, was then called, and deposed to undergoing the experiment already referred to.

The Court then adjourned till Saturday.

SATURDAY.

The whole of the following witnesses deposed to having used the "Excelsior" baking powder in their homes without the least inconvenience or apparent injury to health:—Mrs. Martha Johnson, Port Tennant; Dr. Leckey, Mrs. Kingrate, Mrs. Charlton, Mrs. James, all of Pontypridd; Mrs. Williams, Rhondla; Mrs. Phillips, Treherbert, and Mrs. Agnes Thomas and Mrs. Elizabeth Jones, Blaenrondla.

Dr. Dyke, medical officer of health for Merthyr, was next called, and deposed to having used a preparation very similar to the baking powder in question in his family for a great many years without the least harm resulting.

Cross-examined by Mr. David Lewis: It had only been used in pastry and not in bread.

Mr. David Lewis: I don't know whether you suffer from dyspepsia?

Dr. Dyke: Oh, I'm a martyr to it. I have had the cholera four times. (Loud laughter.)

Re-examined by Mr. Arthur Lewis: He did not attribute the dyspepsia to the use of the baking powder.

Dr. Warburton, surgeon to the Bute Engineering Company, and residing at Treherbert, also deposed to having used some of the baking powder in his family without any injurious effects.

Dr. William Taylor, senior physician to the Glamorganshire Infirmary at Cardiff, and Dr. Vachell, Cardiff, late physician to the Glamorganshire and Monmouthshire Infirmary, both gave similar evidence.

In cross-examination, Dr. Vachell said he was exceedingly pained and surprised that a man of eminence like Dr. Lauder Brunton should have lent himself to guess at the suggestion that bulbar paralysis and other obscure diseases were probably due to the absorption of alumina into the system. Witness admitted that he had not himself made any experiments. He relied upon what he read in the medical journals. The *British Medical Journal* was his Bible. (Laughter.)

Re-examined by Mr. Arthur Lewis: The *British Medical Journal* had published a leading article to the effect that alum baking powders were injurious to health, but in the following week it retracted the statement.

Drs. Latimer and Arthur Davies, both of Swansea, were also called, and bore out the previous evidence as to the non-injuriousness of alum baking powders.

The Court again adjourned till Monday.

MONDAY.

On resuming on Monday morning, Mr. Wynter Blyth was at once placed in the box. Besides holding many qualifications and being the author of numerous works relating to hygiene, he stated that he was medical officer of health and borough analyst of Marylebone, public analyst for the county of Devon, as well as a barrister. In his opinion the baking powder in question, used according to the directions, was not injurious to health. He had for the past twelve months used an alum baking powder similar in constitution to the "Excelsior" baking powder, and had not suffered from any ill effect, nor had any of his family applied to him in consequence of any ailment. He agreed with the previous witnesses that the quantity of hydrate of alumina used in Dr. Morgan's experiment was too great, and also that it being freshly precipitated was not the same as hydrate of alumina baked in bread. Mr. Blyth was cross-examined at considerable length.

This concluded the evidence, and Mr. Brynmor Jones, by permission, again addressed the Court. He said he had distinctly challenged the other side to produce a single instance where the use of this baking powder, either in bread, cakes, or pastry, had been attended with the slightest indigestion or injury, but they had not done so, and, therefore, how could it be said to be injurious to health? Besides, it had been proved that about thirty tons per week of alum baking powder came out from Norwich, and surely that fact alone was a strong argument in favour of the appellant. He would remind the Bench that the words "any substance injurious to health" as contained in the Act were very vague. In construing the Act, he submitted, they could not take those words in the broadest possible sense, or else alcohol and divers other substances which, if mixed up with food, would come within the meaning of the Act.

The Chairman: Do you not think the Legislature deliberately intended to use the widest terms that could be inserted in the Act of Parliament in order to protect the public from any kind of harmful admixture.

Mr. Brynmor Jones replied that he thought the Legislature studiously avoided using medical terms, and contended that the simpler words used did not, therefore, imply that they should be taken in the widest sense. The learned counsel then refuted the theory of the respondent that chloride of aluminium was formed from the hydrate of alumina, or that any injury was sustained from the use of alum baking powders, and went on to refer to the experiments as to the solubility or insolubility of alumina hydrate in the gastric juice made on either side. Supposing they put experiment against experiment, what, he asked, became of the proposition advanced by his learned friend? The only point which he thought any reasonable man could take exception to with regard to the experiment upon Dr. Latimer's coachman was the different dialyser that was used than with Dr. Morgan's experiments. In answer to that, one or two chemical experts had said that the sheep's bladder made a better dialyser because it was more like the natural membrane of the body.

The Vice-Chairman: We cannot accept that, as Mr. Sutton admitted that he tried to get a similar one to Dr. Morgan's, and the only reason he used the sheep's bladder was because he failed to get the other.

Mr. Jones: However, I claim on our experiment the verdict of the Court. Continuing, the learned counsel said that the proposition that chloride of aluminium was injurious was based on the scantiest and flimsiest evidence. Who said it was injurious? Dr. Lauder Brunton. But if they put him forward, he would put forward the evidence of Dr. Richardson and Dr. Luff, who both said it was not. It would not be necessary to go minutely into the question of how far Dr. Brunton's arguments and reasons might be well-founded, because they were admittedly problematical. He (counsel) would beg the Bench to consider his general observations, and asked for a verdict at their hands.

Mr. David Lewis followed with a speech of considerable length, and at the outset dwelt upon one or two points of law as to what was meant by the words "food" and "mixed," contained in the Act, pointing out that, according to the evidence, carbonate of soda had been known to be used as a baking powder both in Scotland and North Wales, while in the "Excelsior" baking powder it had been "mixed" with alum. In the course of his remarks, Mr. Lewis said (1) that the other side had nearly entirely misapprehended the case for the prosecution. Mr. Brynmor Jones had stated that the prosecution contended, amongst other things, that when chloride of aluminium was formed it was absorbed into the system. This they had never said. What they did say was that the chloride formed in the stomach did twofold injury there by irritating the mucous membrane, and interfering with the digesting of fibrin, that when the food got into the large intestine where the fluids are alkaline, the chloride became re-converted into soluble hydrate of alumina and soluble phosphate of alumina, and that a soluble aluminium salt was absorbed into the system. The experiments of Dr. Morgan and Dr. Griffiths were absolutely conclusive, not only in their opinion, but in the opinion of Professor Dunstan and Dr. Brunton, that chloride of aluminium was formed in the stomach when bread made of this baking powder was eaten. Professor Dunstan had, by his four experiments, proved the same thing, and further proved what had never been controverted, that the chloride was re-converted into soluble hydrate in the large intestine—and that in itself was bound to cause the conviction to be upheld, and to establish the case of the respondent—had been proved by experiments, and his evidence upon this point had never been controverted by any single witness for the defence. In fact, no witness had been called to attempt to controvert the statement that the digestion of starchy substances was interfered with to the extent of 27 per cent. Dr. Brunton, whom Dr. Luff described as a man of whom all medical men in England might justly be proud, had stated that whereas it was impossible to say with certainty what was the injurious effect of the absorption of aluminium salts, there was no doubt that such injury would occur, and that possibly some one of the obscure diseases, with whose origin medical science was at present unacquainted, might be due to this very cause. He then dwelt exhaustively upon the evidence, and also concluded by observing that the respondent was entitled to the verdict of the Court.

THE DECISION.

The Bench retired to consider their decision, and at six o'clock, or after about twenty minutes' absence, they returned into Court, when the learned Chairman said: "The Court finds that the appellant unlawfully sold, on December 10th, 1892, a baking powder, which is an article used for food within the meaning of the Food and Drugs Act, mixed with a certain ingredient, to wit, alum, which is injurious to health. The conviction is therefore confirmed and the appeal dismissed with costs."

Mr. Brynmor Jones: Nothing has been said, sir, about costs. You have given against us as I apprehend, but I will only point out to you that a certain case was made by the prosecution at Pontypridd, and the appeal was founded upon that case. It is now common ground between us, as my learned friend has admitted it, that the case which they have made at these Sessions upon this appeal is a very different one. We brought our appeal upon the basis that we should have to meet the evidence given at Pontypridd, but the whole theory advanced by Dr. Lauder Brunton and Professor Dunstan was novel to us. Having regard to that fact, the appeal should be dismissed with costs.

The Chairman: Your client, if he had just paid his 40s. and costs, would have put an end to the matter, instead of incurring the enormous costs of this appeal.

Mr. Jones: I think the sellers of this article have behaved very properly in coming to the rescue of Mr. James. The whole trade is involved. However, I accept your decision, but I thought it my duty to mention it. Another point I wish to take, and this has been practically agreed between my learned friend and myself, subject to your sanction, that a case should be granted upon the following points:—(1.) That there is no evidence that any article mixed with any ingredient or material injurious to health was sold. and that baking powder is not an

article of food within the meaning of the Food and Drugs Act, 1875, section 3. (2.) If baking powder is an article of food, then there was no evidence to show that any ingredient injurious to health was mixed. (3.) That there was no evidence that the baking powder actually sold was injurious within the meaning of section 3.

The Vice-Chairman: The last point we cannot agree to. It is a question of fact.

Mr. Brynmor Jones replied that he submitted it as a point of law, and pointed out that no evidence of any specific injury done had been adduced.

The Vice-Chairman: But if we find it was injurious, there must have been evidence that it must injure to some extent.

Mr. Brynmor Jones: But there never has been!

The Vice-Chairman: But we find it.

Mr. Brynmor Jones then withdrew the third point, and leave to state a case was granted on the remaining two points.

REVIEW.

PUBLIC HEALTH LABORATORY WORK. By HENRY R. KENWOOD, M.B., D.P.H., F.C.S., Instructor in the Hygienic Laboratory, University College, and Assistant to Professor Corfield in the Public Health Department; Late Assistant Examiner in Hygiene in the Science and Art Department, South Kensington, &c.; and RUPERT BOYCE, M.B. (London, H. K. LEWIS, 136 Gower Street, W.C.)

The reviewer rises with a feeling of sadness at the thought that in these times of higher chemical education a book like the one under review can be written or sold. The greater part of it, that to which the following lines apply, is intended to teach and guide those seeking Public Health Degrees. It is written by a medical man who possesses the D.P.H. of Cambridge, and who teaches at an important Metropolitan College. Why cannot medical men teach that which they have specially studied and what they know, instead of attempting to enter into matters of which, as a rule, they have but a smattering of knowledge? What if a chemist were to write on anatomy or on pathology? There have been, and there are, a few brilliant exceptions among medical men, who have mastered one or more departments of chemical knowledge; but the writer of 424 of these 491 pages, is by his own showing, not among these. If he were guilty only of retailing second-hand knowledge correctly, of prolixity, and of vagueness, he might be passed over in silence: but the book before us contains so many grave errors, so many mischievous assertions and faulty statements, that it becomes a duty to expose them and to put the student upon his guard.

Lest we should appear to be unjust to the author, we propose to quote from his work somewhat fully, taking first the chapters which are of special interest to the Public Analyst.

Chalk is stated to be frequently added to milk as an adulterant (p. 283). A fair limit for fat in a pure sample of milk is given at 2.5 per cent. (p. 285), and it is asserted that the matter of a fair limit remains unsettled, but must be so fixed as to include milks which contain even less than 2 per cent. of fat (p. 284). The test for boric acid in milk

given is an almost useless one (flame-coloration). The method of fat extraction as used by Dr. J. Bell is declared to be capable of giving "finer" estimations than those obtained by the use of especially devised fat-extraction apparatus (p. 277). We are informed, in mystic language, that the reduction of fatty and non-fatty solids by the addition of water will afford some clue to any watering, "more especially, however, would this be the case with the fatty solids, since these are not fraudulently added to anything like the same extent as the non-fatty solids."

Butter is stated to be adulterated with *linseed and palm oil* and butter-substitutes must be labelled margarine under the *Sale of Food and Drugs Act*. For the estimation of the saponification equivalent, 25 c.c. of "an" alcoholic solution of potash of undefined strength are to be used. Pure butter "never" yields less acidity for 2.5 grammes of the fat than corresponds to 12 c.c. of decinormal soda solution in the Reichert process, and a correction of 2 c.c. may be necessary, in the author's hands.

Fusel oil is stated to be the chief adulterant of spirits (p. 370), and the *ash* of vinegar, *if acid*, is said to show the addition of mineral acid to the vinegar. Sand is still added to sugar (p. 377). Tin in preserved provisions is best tested for by evaporating the solution suspected of containing tin to a small bulk, adding a drop or two of hydrochloric acid, followed by a few drops of a solution of sulphuretted hydrogen, when, if the metal be present, a yellow or blackish-grey precipitate is formed. These precipitates dissolve in "liquor potassæ," but the best corroborative test "is the expensive one of adding gold chloride to the solution acidified by hydrochloric acid and obtaining the beautiful and characteristic colour known as the purple of Cassius." If a student added to, say, the liquor taken from a tin of preserved fruit, a few drops of hydrochloric acid and the same of sulphuretted hydrogen solution and told us that he could detect tin by the reactions given by our author, even if he used the "expensive" reagent alluded to, we should be inclined to think he was "swerving from the truth."

Aniline colours, we are told, if free from arsenic, are harmless. Has the writer the book never heard of Naphthol-green, Dinitrocresol, Martius-yellow, or other poisonous so-called aniline colours?

The author devotes a large portion of his book to water analysis, and teaches many novelties and processes deserving careful investigation before they are adopted. Lime is estimated by "creating the white precipitate of the oxalate of calcium" by adding oxalate of ammonium, collecting the precipitate, burning it until "the filter paper has been entirely destroyed, the residue being CaC_2O_4 , or the precipitate may be weighed as carbonate"! How this interesting salt, burnt oxalate, may be converted into carbonate we are not informed. No chloride or other salt of ammonium beside the oxalate is directed to be added to the water before precipitation, but the magnesia is nevertheless to be determined in the filtrate, no instruction being given how to treat the ammonium-magnesium phosphate. The silica in water is determined by evaporating 500 c.c. to a solid residue (no acid added

to the water), the residue being "treated with strong hydrochloric acid and then well washed with boiling water." Phosphates are tested for by adding "a few drops of pure and colourless nitric acid and then about twice as much of a solution of the molybdate of ammonium to some of the water in a test tube, boiling well." The author expects to get a yellow precipitate under these conditions, and applies the terms recommended by the Water Committee of the Society of Public Analysts, "traces," "heavy traces," and "very heavy traces" to indications alleged to be obtained under his new method of testing.

For arsenic in water, Professor Kenwood employs the Reinsch test (upon the original water), and elsewhere he gives the original Marsh test (deposition of metallic arsenic from the flame upon a cold piece of china) as a sensitive test, and makes no mention of the, to him, unnecessary refinement introduced by Berzelius. For the determination of the albuminoid ammonia he boils the alkaline permanganate, without making it clear whether he boils the whole litre of alkaline permanganate solution for the preparation of which he gives directions, or only the quantity needed for one determination. In either case we should expect his albuminoid ammonia to be excessive. Professor Kenwood does not appear to understand the oxygen process; he still makes his standard-soap solution for hardness estimations by dissolving ten grammes of Castile soap to one litre; he precipitates magnesia, in presence of ammonium salts, by means of "liquor potassæ" (p. 158); and he confounds nitrates and nitrites (p. 77), both as to testing and in stating that nitrites are largely used as a manure.

We are told that "alum" exists normally in the pure flour, and on four other occasions he uses the term "alum" instead of alumina. This series of errors is corrected in the errata, but we cannot avoid the conclusion that the author, when writing, did not know the distinction between alum and alumina, and that the correction is an after-thought.

To give a full account of the errors contained in this book would require almost a reprint of the work in THE ANALYST; a misstatement is to be found on nearly every page. The few quotations we have given must suffice, but Professor Kenwood's book affords a startling illustration of the perfunctory and inaccurate work which passes muster at at least one University, and of the utter worthlessness of the qualification of D.P.H. as a proof of competency to analyse food or water.

We must condole with Prof. Boyce upon the misfortune to have had his contribution on bacteriological research, which is most clearly written and meritorious, included in this work.

The print of the book is clear, and some of the figures very good, notably a picture of a Winchester quart bottle and a sample hamper.

O. H.

THE ANALYST.

JULY, 1893.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on June 7th, at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hahn occupied the chair.

The minutes of the last meeting were read and confirmed.

It was announced by the Secretary that a letter had been received from the Council of the British Institute of Public Health, cordially inviting Members of the Society of Public Analysts to attend the Annual Congress of the Institute. The Congress would be held this year in Edinburgh, on Thursday, July 27, and the four succeeding days.

The following gentlemen were proposed: As Associates, Mr. Ashworth, assistant to Mr. Smetham, and Mr. Watmough, assistant to Mr. Fairley.

The following gentlemen were duly elected: As Members, Mr. George H. Allibon, Belfast; Mr. Anthony Roche, Dublin; Dr. H. J. Alford, Taunton. As Associates, Mr. C. H. Harrison, Mr. G. E. Scott Smith, Mr. L. K. Boseley.

The following paper was then read by the author

THE SULPHURIC ACID HYDROLYSIS OF BUTTER-FATS.

By Dr. S. RIDEAL.

NOTWITHSTANDING the various improvements which the Reichert process for determining the volatile fatty acids in butter-fat has undergone, there seems to be room for still further modifications.

Many authors have noticed the ethereal odour which is produced on saponifying the fat with potash in presence of alcohol, and pointed out that a possible loss of volatile acids may take place at this stage of the process. In addition to this possible source of error, the formation of carbonic acid from the alcohol, the presence of acid in the alcohol, and the presence or formation of aldehyde, and consequent production of acetic acid, may tend to raise the number of cubic centimetres of alkali consumed in the final titration. The action of the potash on the liberated glycerine has also been only imperfectly studied, and, finally, the time required to remove the last traces of alcohol further militate against the process. Mr. Richmond (ANALYST, 1892, p. 171) has given English readers a good *résumé* of the process and its modifications, but further details on the points just raised will be found treated at length by M. A. J. Zune, in his "*Traité Général d'Analyse des Beurre*."

The idea of substituting for alcoholic potash sulphuric acid as the hydrolysing

agent seems to have been first worked out by Dr. H. Kreis, of Basel (*Chemiker Zeitung*, 1892, 16, 1,394; *Chemisches Centralblatt*, 1893, p. 234), who seems to have argued that the method adopted for the liberation of stearic acid in its ordinary method of manufacture from stearin might be capable of application to butter analysis. He found that butter-fat dissolves in concentrated sulphuric acid at the ordinary temperature, forming glycerine and fatty acids without other products, and that the decomposition takes place much more rapidly than when potash is the hydrolysing agent. The method adopted by him is as follows:

Five grammes of the butter-fat is placed in a half-litre Erlenmeyer flask, and 10 c.c. of concentrated sulphuric acid (sp. g. 1.83) added thereto, and the mixture shaken until clear. The flask is then stood on the water-bath for ten minutes, and 150 c.c. of water added, and the mixture distilled over, using a knee-formed tube without a bulb, but a long 10 cm. stem, into the condenser. The numbers obtained agreed with the Reichert-Meissl method when pure butters were examined, but with margarine and lard higher results were obtained. A genuine butter dissolves clear in the strong acid, but mixtures are usually cloudy at first.

Since the production of the original paper, several foreign investigators have repeated the process, and made modifications in its working.

Since writing this note an abstract of some of these papers has appeared in the last number of *THE ANALYST* (vol. xviii., p. 165).

Dr. J. Pinette (*Chemiker Zeitung*, 1893, 17, p. 395) found that on repeating the above method a considerable quantity of sulphurous acid was produced, and it naturally tended to give variable and high results. To overcome this difficulty he adds strong permanganate solution before distilling until the pink colour is permanent for a few seconds. The sulphurous acid is then destroyed, and the mixture is distilled as in the Reichert-Meissl process until 110 c.c. have come over.

Pinette and Micko have likewise contributed a paper to the *Zeitschrift für Nahrungs Mitteluntersuchungen* (1893, p. 37), in which they repeat that the Kreis method in its original form gives high results.

Their criticism is also supported by Prager and Stern (*Chemiker Zeitung*, 1893, 27, p. 467), who suggest as a method for removing the sulphurous acid to draw a current of air through the liquid until it ceases to smell of this gas. They quote the following analysis in support of their process:

Reichert-Meissl.		Kreis.		Kreis-Prager-Stern.
1. 30.40	...	41.20	...	31.10
2. 24.81	...	33.50	...	28.76
3. 19.90	...	28.30	...	20.68

These authors remove the sulphurous acid by blowing air through the acid before the addition of the 150 c.c. of water.

To these authors Kreis and Shatzmann have replied (*Chemiker Zeitung*, 1893, 31, p. 544; vide also xvii., pp. 395, 468) that the removal of the sulphurous acid is quite unnecessary, provided sulphuric acid of the right strength is employed. Commercial sulphuric acid is too strong, and should be diluted until it equals 91.58 per cent.

H₂SO₄. They further add that they have tried Pinette's modification, and find that it gives low results, and quote the following figures :

	Reichert-Meißl.	Kreis-Pinette.
Butter No. 1 . . .	29.10	18.80
" " 2 . . .	28.60	17.60
Fused Margarine . . .	7.10	5.30

During the last few weeks I have made a series of analyses of butter-fats by the Reichert-Meißl process, and compared the results with that obtained by the use of sulphuric acid, with a view of testing whether the new process is likely to replace it in ordinary work

The sulphuric acid used had a specific gravity of 1.836, and was free from volatile acids. Our earliest experiments gave high results, which were traced to nitric and nitrous acids present in the sulphuric acid employed. Ten c.c. of acid of this strength were added to 2.5 grammes of melted butter-fat, and gave, on shaking, a clear light-brown solution. In all cases sulphurous acid was produced in sufficient quantity to be recognised by its smell. To this mixture, after waiting a few minutes, 100 c.c. of water, free from carbonic acid, were added and the mixture shaken, when the fatty acids clotted out in white flakes. Permanganate solution was then added from a pipette until the solution attained a pink colour, permanent for a few seconds the quantity required being seldom more than about 1 c.c. of a strong solution. The distillation was conducted in the flask fitted with a bulb-tube and delivery-tube to condenser bent at an obtuse angle. Eighty c.c. of the distillate were taken for the titration. Pumice-stone, boiled in sulphuric acid, was added to prevent bumping and on cooling the non-volatile fatty acids solidified to a light-brown cake, which was darker than that of the fatty acids obtained by the ordinary saponification method.

The 2.5 grammes of butter-fat were put into the tared flask when molten, by means of a small pipette with a fine point, and the acid added to the fat.

	Reichert-Meißl.	Sulphuric Acid.
Butter No. 1 . . .	11.50	11.70 and 11.70
" " 2 . . .	13.30 and 13.70	13.70 and 13.70
" " 3 . . .	13.60 and 13.40	14.0 and 14.0
" " 4 . . .	15.10	14.90 and 15.10
" " 5 . . .	15.10	15.70
" " 10 . . .	15.60	15.70
" " 11 . . .	14.50	14.70
" " 34 . . .	13.40	13.70
" " 35 . . .	13.80	14.0
" " 36 . . .	13.50	13.10
" " 37 . . .	14.60	14.70
" " 38 . . .	14.90	14.10
" " 39 . . .	12.60	12.80
" " 40 . . .	14.20	14.80
" " 25 . . .	15.70	15.90
" " 26 . . .	15.80	15.90
" " 27 . . .	15.80	16.00
" " 28 . . .	15.00	15.00
" " 29 . . .	15.30	14.50
" " 30 . . .	15.60	15.90

Mean 14.42 for Reichert-Meißl,
leaving out No. 1.

Mean 14.68 for Sulphuric
Acid, leaving out No. 1

Here also are a few results which have been obtained with margarines and lards:

		Reichert-Meissl.	Sulphuric Acid.
Margarine No. 1	...	80	1.0
" " 2	...	1.50	1.70
" " 3	...	1.50	1.60
" " 4	...	1.10	1.20
Lard No. 1	...	30	40
" " 2	...	30	30 and 40.

The results for margarine, and also for the genuine butters, seem to point to the new method giving slightly higher results than the standard Reichert-Meissl.

I have to thank my assistant, Mr. Bult, for some of the analytical results which are quoted in this note.

DISCUSSION.

Mr. Alfred H. Allen had been struck with the descriptions of the various modifications of the sulphuric acid process as they appeared in the proof-sheets of *THE ANALYST*, and the result was that he instituted one or two experiments upon Pinette's method, in which he used sulphuric acid, and then oxidized the sulphurous acid formed by permanganate. He did not dilute the sulphuric acid, as was now recommended by Schatzmann and Kreis, who reduced it to 1.83 specific gravity, which was almost as great a density as that of the strongest sulphuric acid; but, as had been pointed out, this meant an addition of one-tenth of the volume of water to the strong acid. It was, therefore, necessary to be careful in cases where only the gravity was mentioned. To speak of strong sulphuric acid without specifying the gravity was misleading. His experience, on the few experiments he had made as to the amount of permanganate required, had been very different to Dr. Rideal's. He worked with 5 grammes of butter fat, added 10 c.c. of sulphuric acid diluted, and added a 2 per cent. solution of permanganate, in quantity sufficient to make the mixture pink for a few seconds. The conditions prescribed by Pinette were adhered to, perhaps too rigidly, and consequently a much smaller fraction of the liquid was distilled than in the Reichert-Wollny process. The results did not at all agree with those by the Reichert process, being considerably higher than the latter; but he should certainly try the process again with weaker acid, and under the conditions prescribed by Dr. Rideal.

Mr. Richmond suggested that if, instead of using potassium permanganate, either potassium bichromate or hydrogen peroxide—which were not, as far as fatty acids were concerned, such strong oxidants—were used, the results obtained might come out better. The Reichert process did not satisfy all requirements of the analyst. It was not possible, by means of that process, to detect adulteration with certainty in all cases; and it seemed highly desirable that, instead of attempting to make improvements on this process, analysts should set to work in other directions. There were a good many fields in which he believed information could be obtained which would enable them to get much closer to the detection of adulteration than at present. For instance, he thought there was a great deal to be done by a more detailed examination of the insoluble fatty acids, instead of ascertaining only their

total weight. There were a good many figures which could be obtained on these without much trouble; for example, the saponification equivalent, the iodine absorption, and the freezing-point of fatty acids. The ratio between these would lead to much information. Much might be done in this direction, though it must be remembered that Mr. Hehner had shown that even the separation of the oleic series from the saturated series was by no means easy. Then the solubilities of butter in certain menstrua was useful; in France Bochaire and Honzeau had proposed methods which promised well. The day of single methods was past, and the Reichert process, though it had done good service, could now only be looked on as one link in the chain of evidence.

Mr. Hehner, replying to Mr. Richmond, said that he had every hope of being able before very long to bring the results of a lengthy investigation before the Society, in which he would deal with the suggestions of Mr. Richmond concerning butter-analysis. As regards Dr. Rideal's paper, he might state that some years ago he had also made experiments upon the hydrolysis of butter-fat with sulphuric acid, but could not obtain satisfactory results. It appeared to him that at present the method did not give results which were comparable with those yielded by the Reichert method. In the latter the alkali employed for saponification was neutralized with sulphuric acid, care being taken to have no free sulphuric acid, but only acid sodium sulphate in the liquor to be distilled. On the other hand, by using 10 c.c. of strong sulphuric acid for hydrolysis, none of which was neutralized, it followed that towards the end of the distillation a very strong solution of sulphuric acid was obtained, and consequently there was great risk of sulphurous or some other volatile acid being formed. The sulphuric acid method was inapplicable for estimating the insoluble fatty acids, because the sulpho-oleic acid formed yielded oxy-oleic acid on dilution of the solution, and this latter had a different equivalent from that of the fatty acids obtained by ordinary saponification. He would further point out that, while Dr. Rideal had used 2.5 grammes of fat, with 10 c.c. of acid and 100 c.c. of water, he had the same amount of liquid as was used in the Reichert-Wollny process with only half as much fat, or double as much liquor, as was required for the Reichert process. It was not to be expected, therefore, that the results of the methods could be identical. Dr. Rideal appeared to have, by a happy chance, obtained figures identical with the Reichert figures; but, the conditions being entirely different, the correspondence could be nothing more than a coincidence. He thought that it was exceedingly interesting to have the question investigated from a scientific point of view; but nothing could be much simpler than the method now generally adopted, while the results of the Reichert and Wollny processes were remarkably constant and accurate. It should also not be forgotten that while butter-fat, when mixed with sulphuric acid, evolved but little heat, and only suffered slight decomposition, margarine, which often contained much vegetable fat, would in many cases undergo a much more profound decomposition when acted upon by strong sulphuric acid, accompanied by the evolution of large amounts of sulphurous acid. The process, therefore, was not applicable to anything but pure butter, or to margarine which contained little or no vegetable oil.

Dr. Rideal, in reply, said he did not think there were any questions which he was expected to answer. He quite agreed with Mr. Hehner's remarks as to the quantity of water he had used in his process; but he (Dr. Rideal) wished to use $2\frac{1}{2}$ grammes, and he wished to follow as far as possible Pinette's recommendations, and therefore he had tried in his first experiments the addition of 100 c.c. of water, and then distilling off 80 c.c.; and, finding that the results did agree with the ordinary process, he continued using that quantity. His original idea was, that, as he was dealing with a quantity of strong sulphuric acid, it must be kept dilute. He had not tried any other oxidizing agent. He was glad that Mr. Allen had tried the process, and he hoped that a good many others would do the same.

The Chairman exhibited three photographs of microscopic preparations which had been sent by Messrs. H. B. Stocks and James G. Ross, of 25, Ferndale Road, Liverpool, showing the difference in the crystals of genuine lard, and lard adulterated with beef-stearin obtained by the Barfield method. As the crystals are obtained and examined in ether, the slides cannot be kept for more than a few seconds. Attention was called to the desirability of having a permanent record of these and other samples of food adulteration, since these might be extremely useful in court cases. Messrs. Stocks and Ross expressed their willingness to supply copies of these photographs, or to take photographs of microscopic preparations of any adulterated samples which members might have before them, and of which they wished to keep a record. No price was mentioned.

Papers on the following subjects were also read: "Notes on Rice-Oil and Maize-Oil," "On Rapid Saponification for Analytical Purposes," by Mr. Alfred Smetham; "On Ginger—with special reference to discrimination between genuine and 'exhausted' specimens," by Dr. Bernard Dyer and Mr. J. F. H. Gilbard.

(Conclusion of the Society's Proceedings.)

POINTS IN THE ANALYSIS OF CONDENSED MILK.

By H. DROOP RICHMOND AND L. K. BOSELEY.

(Read at the Meeting, May 3rd, 1893.)

In an analysis of condensed milk the following determinations are desirable:

Total solids, fat, ash, milk sugar (cane sugar), casein, albumin, total proteids (Bitthausen), and acidity as lactic acid. A determination of total nitrogen is also useful.

The plan we adopt is to weigh out from 15 to 30 grammes of the condensed milk into a 100 c.c. flask, to make up to 100 c.c. and weigh the total; definite weights of the diluted milk will then represent definite weights of the original.

Total Solids and Ash.—Five grammes of the diluted milk are weighed into a basin packed with asbestos, and dried for about 12 to 18 hours (to constancy); the residue is afterwards incinerated (preferably in a muffle).

Fat.—Five grammes are placed on an extracted coil, and the fat estimated by Adams' method; as a check the fat is extracted from the Ritthausen precipitate. The two methods, with care, agree well.

Milk Sugar and Cane Sugar.—These cannot be estimated by the polariscope, as the milk has been heated (ANALYST, xviii. 141). Stokes' and Bodmer's method (ANALYST, x. 62) is suitable, but probably not very reliable (*cf.* Hehner, ANALYST, vi. 218); it is better to weigh the copper oxide reduced before and after inversion with citric acid.

The use of invertase, as proposed by Kjeldahl, would probably be suitable, but we have no experience of this method (*Z. Anal. Chem.*, xxii. 558).

It is a good plan to estimate the reducing powers of a known weight of pure milk or cane sugar, about equal to that in the quantity of condensed milk taken, and to calculate direct from this; in this way all corrections and factors are avoided.

Shenstone's method (ANALYST, xiii. 222) is evidently unreliable, as it involves the use of the polariscope, though in some cases we have obtained satisfactory results with this instrument. We have taken the liberty to correct his sugar figures by dividing the total polarization by 1.042, and they stand then as follows:

	1.	2.	3.	4.	5.	6.	7.
Water . . .	30.3	24.8	26.8	28.0	26.4	25.6	26.8
Fat ...	4.7	4.7	4.7	4.0	11.5	10.4	10.6
Proteids ...	12.6	12.4	12.4	12.1	12.6	10.7	11.1
Ash ...	2.1	2.4	2.3	2.4	2.1	1.9	1.9
Milk Sugar ...	15.3	15.7	14.6	14.9	14.4	14.5	14.2
Cane Sugar . .	33.2	36.6	37.8	36.6	28.2	31.8	30.4
Total ..	98.2	96.6	98.6	98.0	95.2	94.9	95.0
Error ...	1.8	3.4	1.4	2.0	4.8	5.1	5.0

We believe it to be quite legitimate to correct the sugar figures thus, as in no part of his paper does Shenstone give even a hint that he departed from Vieth's mode of using the polariscope; one of us (ANALYST, xvii. 222) has already shown this correction to be necessary, and the error introduced by its neglect is large in condensed milk.

Casein and Albumin.—We use Sebelien's method (*Zeit. Physiol. Chem.*, 13, 135)* without modification. The Kjeldahl determinations were made with the addition of a little copper oxide to the sulphuric acid; no sulphide is necessary in the distillation; we find it an advantage to use 30 c.c. of sulphuric acid for the casein precipitate.

* *Sebelien's Method for the Estimation of Casein and Albumin in Milk.*—To 10 grammes of milk add 20 c.c. of a saturated solution of magnesium sulphate and crystals of magnesium sulphate, as long as they dissolve on agitation; a small excess of crystals will not do harm. To obtain correct results, the milk must be as neutral as possible, and the magnesium sulphate free from sodium sulphate (commercial Epsom salts often contain this impurity). The mixture is then allowed to stand for some hours, poured on to a filter, and washed with a saturated solution of magnesium sulphate. The washing can be performed in five to six hours if the filter be not allowed to run dry. The filter, with the precipitated casein, is treated by Kjeldahl's method, using 30 c.c. of sulphuric acid. The nitrogen found, multiplied by 6.37, equals the casein. The filtrate is then diluted, and a few drops of tannin solution (Alumen's[†]) or phosphotungstic acid solution, added until precipitation is complete. The precipitate is collected on a filter, slightly washed, and treated by Kjeldahl's method. The nitrogen found multiplied by 6.37 gives the albumin.

[†] Alumen's solution, see ANALYST, xvi., p. 86, footnote.

Ritthausen's Method.—This method is a dangerous one, as the pitfalls are many and not very apparent. It is founded on Ritthausen's observation that the copper oxide compounds of albuminoids are insoluble in water. It is usually directed to use for 10 c.c. of milk 5 c.c. of 6.5 per cent. solution of crystallized copper sulphate, and to nearly neutralize the acid set free; we are of opinion that this quantity of copper solution is excessive, and in the neutralizing a quantity of hydrated oxide or basic compounds are thrown down, and the whole of the matter volatile on ignition is not driven off at 100° – 130° , which swells the apparent proteids.

We are inclined to doubt the liberation of sulphuric acid, which has a very large heat of neutralization, by an albuminoid; we may mention that Béchanp (*Mémoires prés. à l'Acad. Sciences*, xxviii. 3, 98) is of opinion that casein and albumin exist in milk as salts, and in confirmation of this view that Söldner (*Landw. Versuch Stat.*, 35, 351) has found that in milk there is an alkali in combination as organic salt over and above that required to neutralize Henkel's citric acid (*Molkerei Zeit.*, 2, 259). Milk begins to taste sour and curdles on boiling when the acidity to phenolphthalein is equal to about 0.4 per cent. lactic acid, an amount but slightly greater than that found by Söldner to be necessary. Acting on this view, we have tried neutralizing the milk before adding copper sulphate, with very satisfactory results. We are, however, of opinion that the results thus obtained have a tendency to be a little low, on account of the oxidation of the phosphorus of the proteids to phosphoric acid, which remains combined with the copper on burning. Our Ritthausen results are about equal to the casein and albumin determined separately, and a trifle lower than the total nitrogen multiplied by 6.37. Lately Carcano has published figures showing that Ritthausen's original method is a little higher than the total nitrogen $\times 6.37$ (*ANALYST*, xvii. 134).

Our procedure is as follows: Dilute 10 grammes of the diluted milk with about 200 c.c. of water, add a few drops of phenolphthalein solution, and neutralize with dilute caustic soda solution, add 2–2.5 c.c. of copper sulphate solution (*as above*), allow to settle, wash about five times by decantation through a tared filter, and then wash on the filter, spreading the precipitate over the filter, dry slightly in the water oven, extract the fat, dry at 130° , burn and subtract the ash.

This modification of Ritthausen's method gives good results with all milk products except whey, which contains albumoses produced by the action of rennet (chymase), which one of us has called chymo-albumoses (*Chem. News*, 67, 132). Neumeister has shown that deutero-albumose obtained during digestion studies by Kühne and Chittenden's method (*Zeit. Biol.*, 23, 381) gives a soluble copper salt, and we have found that the Ritthausen precipitate with whey does not correspond with the total nitrogen multiplied by 6.37. In a mixture of milk and whey in about equal parts we have found about 0.3 per cent. of albumoses precipitated neither by copper sulphate (by our modification) nor by magnesium sulphate, and boiling after slight acidification (to remove albumin); in Sebelien's method albumoses would be estimated as albumin, so that if whey were mixed with milk the Ritthausen precipitate would be less than the casein and albumin, and the albumin would be abnormally high. Chymo-albumoses can be precipitated, however, as very basic copper salts.

Lactic Acid.—We calculate the total acidity as lactic acid.

Of the condensed milks we have analyzed, we may mention one called "The first Swiss brand of unsweetened condensed milk."

This has been analyzed by Faber (*loc. cit.*), and an analysis by a chemist named Goodfellow is given away with the tin.

	R. & B.	Faber.	Goodfellow.
Water ...	63.47	62.97	61.3
Fat ...	10.22	10.67	11.7
Sugar ...	13.94 (diff.)	14.55 (diff.)	14.3
Casein...	9.81	9.24	9.1
Soluble Albumin	0.49	0.32	1.5
Ash ...	2.07	2.25	2.1
	100.00	100.00	100.0

The amount of sugar by polarization was found by us to be 11.5 per cent.

The only important difference in the three analyses is in the soluble albumin, and as great stress is laid upon this large amount in Goodfellow's report, we can only suppose that this chemist is of opinion that his determination represents the amount of soluble albumin in the milk as sold. We can only suppose that he analyzed the milk before sterilization by heat, or that he used some incorrect method of analysis.

Our analysis and Faber's are as concordant as two samples analyzed some years apart could be expected to be. Both the low polarization and the low albumin show plainly that the milk has been heated.

A large class of condensed milks in the market are labelled "skimmed" milk. As these contain "separated" milk, we are of opinion that the label does not comply with the requirements of the Sale of Food and Drugs Act, 1875, section 10. *Skimmed* milk distinctly means that the milk has been set, and the cream that rises has been skimmed off, and this should contain about 1 per cent. of fat.

Separated milk is milk from which the cream has been removed by a separator, and contains about 0.25 per cent. of fat.

The purchaser has a right to expect about 3 per cent. fat in a milk concentrated to one third, as most of these are, and these brands frequently contain much less, though called "skimmed milk."

In our opinion the two words are not synonymous, and are not even so in loose dairy phraseology. It is true that separated milk is often spoken of as "skim" milk, but not to our knowledge as "skimmed." Were these brands labelled as "skim" milk, in our opinion a conviction would be nearly impossible if the use of separated milk were proved; but as they bear distinctly the word "skimmed," which is frequently accompanied by the jesuitical statement that a portion of the fat has been removed, our opinion is that the use of the word "skimmed" might be held to constitute a false label under the Act. It is also possible that the description on the label of many brands, that "a small quantity of cane sugar is added," would also be held to constitute a false label under the Act if the amount of cane sugar exceeded the amount of other solid constituents.

Analyses of such a sample of milk (A) and of a good quality of condensed milk (B) are subjoined:

	A.	B.
Water ...	29.05	24.86
Fat ...	1.28	11.28
Proteids ..	10.63	9.36
Ash ...	2.33	2.13
Milk Sugar	14.91	13.97
Cane Sugar	40.07	38.81
	98.27	99.91

Assuming that these milks are concentrated in the proportion 1 : 3, the original composition of the milks was :

	A.	B.
Fat43	3.76
Solids not Fat	9.29	8.49

The "skimmed" milk is probably badly separated milk, while sample B contains a good proportion of cream.

NOTE ON THE DETECTION OF ADULTERATION OF FRESH MILK BY DILUTED CONDENSED MILK.

BY H. DROOP RICHMOND AND L. K. BOSELEY.

(Read at the Meeting, May 3rd, 1893.)

SOME five years ago a Dairy Farmers' Association offered a gold medal for the solution of this problem. Their chemist advised them that it was not possible, and he had no hope of its ever being done. The medal was not awarded.

Faber (*loc. cit.*) has devised a method of detecting the adulteration by the diminished amount of soluble albumin in heated milk, and we have been able to amply confirm his results.

We have found the albumin to vary from 0.41 to 0.45 per cent. in fresh milks, which is within Faber's limits, 0.35—0.45. The reduction of the rotary power of the milk sugar will also be of great assistance. We would recommend analysts to estimate the milk sugar by the polariscope, and if it falls below 52 per cent. of the solids not fat to make careful gravimetric determinations of the milk sugar by Fehling's solution; to satisfy themselves that the deficiency is not due to lactic fermentation, by acidity estimations; and to estimate the soluble albumin by Sebelien's method.

A discrepancy between the gravimetric and polarimetric estimations of the milk sugar will afford considerable evidence that the milk has undergone heating, and if the soluble albumin is also low the evidence will be almost conclusive.

The limits in fresh milks may be taken as albumin 0.35 per cent., milk sugar and lactic acid 55 per cent. of the solids not fat, and the gravimetric and the polarimetric estimations of milk sugar will not differ more than 0.15 per cent.

The diphenylamine test for nitrates would also afford strong corroborative evidence if impure well water (common on farms) had been used. We operate as follows: Curdle the milk by heating and adding the least possible excess of acetic acid; dissolve a little diphenylamine in pure sulphuric acid (1 c.c.), and allow a few

drops of the serum to flow over the surface. A blue colour is characteristic of nitric acid; we have obtained the reaction in a milk to which 5 per cent. of water has been added.

DISCUSSION.

Mr. Faber was very much interested in seeing that Messrs. Richmond and Boseley had taken up the method of analysis which he had some years ago brought before the Society, and which had been proposed by Sebelien. He (Mr. Faber) had only time on that occasion to make a few estimations of albumin in milk—boiled or fresh. He quite agreed with the authors of the paper that the amount of albumin found by Sebelien's method must be absolutely unreliable—that is to say, if the latter meant soluble albumin. He thought, even if Sebelien meant all the albumin present, either precipitated or soluble, the figures were somewhat overstated.

Dr. J. A. Voelcker thought it undesirable to draw a distinction between skim milk and skimmed milk, because it seemed to him that such a distinction would certainly never hold good: not only on account of the similarity of the words, but because all minute descriptions of this kind seemed to him impracticable. As to the test based upon the detection of nitrates and nitrites in the water used, it was quite a frequent occurrence to find water which contained no nitrates at all, and then the test would be useless.

Mr. R. Bodmer, referring to the determination of sugar, wished to know if the citric acid inversion was the only portion of the Stokes and Bodmer process used by them. Many years ago he analyzed a sample of Swiss condensed milk, and had found that it was absolutely free from cane sugar. He would like to know whether the authors of the paper had found the same.

Mr. H. Droop Richmond: Yes, in some cases.

Mr. Otto Hehner expressed the pleasure he felt at seeing Mr. Boseley (a former pupil of his) come forward with a paper. He was glad that the authors had been more successful with the diphenylamine test than he (Mr. Hehner) had been. His difficulty had been that sulphuric acid alone produced an objectionable colour with the milk direct, or with the residue obtained by extracting the milk-solids with alcohol and evaporating the latter. He observed that Mr. Richmond allowed the milk to be tested to flow over the sulphuric acid, avoiding mixture. He noted that the authors drew a distinction between skimmed milk and separated milk. He had learned with surprise that this question had arisen in a court of law, in the prosecution of a vendor of an article labelled "Skimmed Condensed Milk." The sample in question was plainly and honestly labelled in large letters "Skimmed Condensed Milk," the Analyst who gave evidence stating that the contents did not consist of skimmed milk, but of separated milk. The vendor was fined. He (Mr. Hehner) thought that the point was not worthy of the consideration of Public Analysts, who had far more important matters to attend to. The difference between milk that had been hand-skimmed and such that was machine-skimmed was so small that it was not worth talking about in an article like condensed milk, or to make a point of it in a prosecution under the Sale of Food and Drugs Act. Certainly, in most cases, machine-skimmed milk contained a little less fat than hand-skimmed milk, but, on the other hand, separated

milk was quite fresh, while hand-skimmed milk was more or less sour. There were, at the same time, very numerous analyses of skimmed milk on record which showed that often hand-skimming was at least as effective to remove the cream as machine work, and the Analyst could not, therefore, be in a position to prove by determination of the fat alone what method of skimming had been employed, unless, indeed, the whole of the fat were removed, which was not the case in the sample subject to prosecution. No two separators worked exactly alike, and even the same machine gave different results on different days. Therefore, if the Analyst concerned carried out his contention to the logical extent, he would have to insist upon the name of the machine used being stated, as well as the number of revolutions of the separator. His decided opinion was that, due notice having been given by a plain and honest label that the fat had been removed by skimming, the purchaser was not prejudiced, no matter whether the skimming had been done by hand or by separator, or more or less completely. The purchaser had no right to expect in skim-milk any fat at all, and any that was left in the skim-milk was really a present to him and a loss to the manufacturer of the condensed milk. No ordinary purchaser expected any fat in skim-milk at all. He thought the point was a mere quibble, and not exactly fair to the merchant who tried to conduct his business honestly. At the present time there was no consensus of opinion, even among experts, in this matter, and unless by common consent a distinction was made between skimmed and separated milk, it was not right to spring such a point in a court of law upon the vendor. He was strongly opposed to the sale of skimmed condensed milk, unless due notice was given to the buyer, but such notice having been given plainly and unmistakably, it appeared to him quite immaterial how and to what extent the skimming had been effected. The absurdity of the position was made plain by the attempt of the authors of the paper to make a distinction between skimmed milk and skim-milk.

Mr. Cassal disagreed with Mr. Hohner. He could not admit that because Public Analysts had a number of other things to deal with, therefore this matter should be left alone. The Public Analyst had to deal with the things which were submitted to him. It happened that certain public Authorities were in the habit of deciding what articles should be analyzed, and it was not advisable for a Public Analyst to object to that course of action. The persons responsible for the taking of samples in such cases as those referred to were the Committee of the Authority. It invariably threw suspicion on a public officer if he raised objections to any course which was desired by a Committee or a Board. The case referred to was only one among many. These articles were generally sold under the name of "Condensed Milk," and in some corner of the label, printed in small letters, there was a statement containing the word "skimmed," the word being introduced on some other part of the label than that containing the principal title. Statements of that sort no one could possibly defend. If a condensed milk was practically devoid of fat, that fact should be stated fully on the label, and should form part of the title of the article. The use of the word "skimmed" was not a sufficient disclosure of the real nature of such an article, and the label that Mr. Hohner had alluded to did not contain a sufficient disclosure. Why was it that a certain class of vendors desired to adhere to the use of the word "skimmed" and would not use the word "separated," which

accurately disclosed the nature of the article? The only answer was that the word "skimmed" conveyed a more favourable impression, and that, in fact, the article so described would be thought better than it really was. Did anyone suppose for a moment that the manufacturers of these articles employed an army of persons to hand-skim their milk before they condensed them? It was a matter of common knowledge that these articles were produced by means of separators, and that they could only be so produced. When Mr. Hehner said that there was but very little difference between the percentages of fat found in milk that had had cream removed by hand and in milk treated by a separator, he forgot also to point out that it was possible to get any results one liked with a separator. It depended on how far it was worked; and the comparison which he made between the percentages of fat were entirely beside the mark, because, in the first place, the figures referred to had been collected from all sources—they were not authenticated, and there was no doubt that many had been obtained by obsolete processes and unreliable persons; and, in the second place, the matter in hand was the comparison between legitimately hand-skimmed milk and milk which had been almost completely deprived of fat, a result which had unquestionably been attained by means of a separating machine.

Mr. Allen quite agreed that the point which had been raised was one of very great interest to the Society of Public Analysts; and the opposite views taken by Mr. Hehner and Mr. Cassal showed how much need there was for discussion. A point which had not been referred to was the misleading statement to be found on the labels of many brands of condensed milk respecting the amount of water required to be added. This was often very greatly overstated, with the consequence that the consumer had milk of far less nourishing power than he supposed.

Mr. E. J. Bevan said that he did not agree with Mr. Hehner, and not altogether with Mr. Cassal. It might be true, as Mr. Hehner had said, that a large number of so-called separated milks certainly showed a higher percentage of fat than skimmed milks. Everything depended upon the way in which one operated. But taking the average composition of separated milks and the average composition of skimmed milks, there was not a shadow of a doubt that the proportion of fat removed in the case of the separated milk was much greater than in the other case. This went a great way towards showing that there was a definite distinction between the two. Some of the samples labelled "Condensed Skimmed Milk" which he had received contained a very small proportion of fat, so small that he could have no doubt that they had been treated in a separator.

Dr. Voelcker hoped that Public Analysts would not attempt to draw distinctions between so-called skimmed and separated milks. He regarded skimmed or separated milk as milk from which the fat had been removed, and in his opinion it was a matter of accident as to how much had been removed. If it could be said that milk always contained a definite quantity of fat when skimmed by hand, and another definite quantity when separated by machine, then clear distinctions could be drawn, but not otherwise.

Mr. Faber said that separated milk contained an extremely low amount of fat, which would not exceed from .1 to .3 per cent. in well-managed dairies. If milk had

to be hand-skimmed before it commenced turning sour, so as to be fit for condensing, it would be found that the skimmed milk contained sometimes as much as 2 per cent. of fat, very often over 1 per cent. He considered that there was a great deal of difference between hand-skimmed milk and separated milk. There was a point in favour of separated milk which might be mentioned, and that was that by separating, not only was the fat removed, but the impurities also to a very large extent. On the inside of the separator bowl an aggregation of very dirty slimy black matter was formed, which contained almost all the dirt present in the milk, and which was thus kept out of separated milk. The value of skimmed milk, as a nourishment, varied to a certain extent with the contents of fat. With regard to the label, if it merely stated that a portion of the fat had been removed, and the amount removed was found to be something like 95 per cent., then he considered it a misleading label, but if the contents of the tins were described on the label as "Skimmed Milk," then he thought this was a sufficient disclosure.

Mr. Cassal thought that Dr. Voelcker's remarks were much to the point, as they admitted the accuracy of his (Mr. Cassal's) contention. Dr. Voelcker had said that the amount of fat left by hand-skimming was uncertain. He had also said that the amount of fat which might be left in milk after using a separator was uncertain. But there was one thing which was certain, and which he understood that Dr. Voelcker and Mr. Hehner admitted without reserve, and that was that when there was practically no fat at all, when 90 per cent. of it had been abstracted, then there was no doubt whatever that the abstraction had been effected by a separator. This was all that he (Mr. Cassal) required. Mr. Faber had published in *THE ANALYST* some years ago a paper in which he advocated the use of the term "separated" for these articles, and in which he said that the word "skimmed" did not afford a sufficient description of them.

Mr. Faber admitted that he had in the paper referred to said that the term "condensed separated milk" would be a more correct description than if it was merely mentioned that "part of the cream" had been abstracted, but he had not used the word "separated" as against the word "skimmed." The difference was between printing on the label in large letters "Skimmed Milk" or "Separated Milk," and of calling the milk with such a name as, for instance, "Goat Brand," and then in small letters adding that "part of the cream" has been abstracted.

Mr. Cassal gathered, from what Mr. Faber had just said, that he adhered to his original recommendation, and that he thought it would be better for the public if these articles were described by their correct name, as "Condensed Separated Milk."

Mr. Allen said he had been struck with the ingenious and powerful manner in which the arguments had been put on both sides. With reference to the whole subject, he would point out that the Government had promised a Select Committee to consider Dr. Cameron's Bill now before the House of Commons as soon as it had passed its second reading. In view of the fact that the Government appeared to be desirous of dealing with the subject of adulteration, although they could not do it very thoroughly on the basis of such a Bill as Dr. Cameron's, it was important that Analysts should be agreed as to how articles should be labelled. He was

afraid that there were not very many people among the lower classes who knew the meaning of the words "separated" milk, and, for his own part, he preferred the term "skimmed" milk. He thought "skimmed milk" covered the whole ground and *included* separated milk, which might be regarded as mechanically skimmed milk. There might be less fat in the mechanically than in the hand skimmed milk, but it would be difficult to establish a definite distinction between them which would stand critical examination. He thought the distinction attempted to be drawn between the words "skim" and "skimmed" was a quibble. They ought to base their action on much broader grounds than these, which were objections only worthy of lawyers. With regard to the estimation of nitrogen in the copper precipitate obtained by Ritthausen's method, he thought that if the precipitate—including the filter-paper, if they liked—were treated by Kjeldahl's process, a much more reliable result would be obtained than by igniting the precipitate and regarding the loss as proteids.

Mr. Richmond, in reply, said that, with regard to Mr. Allen's suggestion of estimating the nitrogen in the Ritthausen precipitate, he had sometimes done so; it seemed to be a more satisfactory method of estimating proteids by actual weighing than by calculation from the nitrogen; the percentage of nitrogen in casein was not known with absolute accuracy, Hammarsten giving 15.65 per cent. and Chittenden and Painter 15.9 per cent., and the error of the factor would be probably as great as the error of weighing. A remark had been made to the effect that in the adulteration of fresh milk by condensed skimmed milk it was not reliable to depend on the presence of nitrates. In their paper they had brought this forward as affording further and independent evidence of adulteration; if nitrates were found it was very strong, and, in fact, the only direct, evidence that the milk had been mixed with water (containing nitrates). Mr. Hehner had asked if they had examined samples of milk which had been purposely adulterated with diluted condensed milk. They had not done so. In a diluted condensed milk they had found a deficiency of 0.8 per cent. of sugar. This paper was not intended as an exhaustive one, it was only called a "note," and they thought that the evidence would induce others to make experiments. The chief part of the discussion had turned upon what they (the authors of the papers) considered a somewhat unimportant point—whether "skimmed" and "separated" milk were synonymous terms. Mr. Boseley and he held no very strong opinion on this point, and had introduced it for the sake of eliciting the opinion of the Society; the subject had some practical interest, as cases had lately occurred. They had stated subsequently that if the words "Skim Milk" were used on the tins, it might be difficult to obtain a conviction, but between "skimmed" and "separated" milk there was a difference. He observed the point was not seen by some. There were two distinct things—skimmed milk and separated milk. Two statements to the contrary had been brought before the meeting, *i.e.*, König's analysis and Dr. Voelcker's experiments at shows. He (Mr. Richmond) did not think that the results given by a machine fitted up at a show could be taken as a fair specimen of what the machine could do, as it was worked frequently under unsuitable conditions, and by operators whose minds would be occupied by details unconnected with the working of the machine. König's

figures were extracted from all kinds of sources, and the majority of the determinations made by methods now admitted to be inexact. With regard to the extremely low percentage of fat sometimes found in hand-skimmed milk, that low percentage could be obtained if desired, but it could not be done on a commercial scale. If one considers that the milk in the tins is always fresh, he thought there could be no shadow of doubt that skim milk would contain more fat than separated milk. Personally, he could echo a remark of Mr. Hehner's, that it was not a serious infringement of the Act, and that there were other adulterations which were more in need of being stopped.

VINEGAR.*

BY ALFRED H. ALLEN, F.I.C., AND C. G. MOOR, B.A., F.C.S.

RECENT prosecutions for the sale, as vinegar, of an article consisting essentially of acetic acid obtained by the distillation of wood, have caused pharmacists generally to consider their position with regard to such an article, and to inquire what is properly to be understood by the term "vinegar."

Ordinary dictionary definitions of technical products are not unfrequently unreliable, but it is nevertheless of interest to note the description of vinegar given in some of our leading dictionaries and encyclopædias.

The "Imperial Dictionary" gives the following definition and description of vinegar:

"Dilute and impure acetic acid, obtained by the vinous fermentation. In wine countries it is obtained by the acetous fermentation of inferior wines, but in this country it is usually procured from an infusion of malt which has previously undergone the vinous fermentation. Vinegar may also be obtained from strong beer, by the fermentation of various fruits, or of a solution of sugar mixed with yeast; in short, all liquids which are capable of the vinous fermentation may be made to produce vinegar."

The "Encyclopædia Britannica" states that—

"Vinegar is a dilute form of acetic acid, having a flavour that varies according to the source from which it is obtained. . . . The nature of acetous fermentation, and the *rationale* of the processes by which vinegar is prepared, are explained under 'Fermentation'; and the acetic acid obtained by the destructive distillation of wood is dealt with under 'Tar.' Here we have only to deal with the various kinds of vinegar used for table, medicinal, and other household purposes."

In the first edition of Watts's "Dictionary of Chemistry" vinegar is classed under the separate heads of "Wine Vinegar," "Malt Vinegar," "Distilled Vinegar," and "Wood Vinegar." In the new edition there is no distinct definition of vinegar. Under the preparation of acetic acid is the following passage:

"Under the influence of ferments.—This is the ordinary process of making vinegar from alcoholic liquids, wine being generally used for the purpose in France and Germany, and malt in England."

T. H. Thorpe, in his "Dictionary of Applied Chemistry," vol. i., states that—

"In all processes for the manufacture of vinegar advantage is taken of the oxidizing action of the vinegar-fungus already described; the souring of wines and other alcoholic liquids is due to this organism, the germs of which are always present in the air, and are deposited, and grow in any suitable medium."

A. H. Allen, in his "Commercial Organic Analysis," vol. i., states that—

"Properly speaking, vinegar is a more or less coloured liquid, consisting essentially of impure dilute acetic acid, obtained by the oxidation of wine, beer, cider, or other alcoholic liquid. Sometimes the term is improperly extended to pyroligneous acid or 'wood vinegar,' while acetic acid is called 'distilled vinegar.'"

S. P. Sadtler, in his "Industrial Organic Chemistry," p. 231, says:

"Only such materials will be considered here as give rise to a vinegar by the normal acetic fermentation. The manufacture of acetic acid and technically important acetates will be spoken of later, under pyroligneous acid, as derived from the destructive distillation of wood. The materials referred to as furnishing vinegar under the influence of the acetic fermentation are—first, wine; second spirits; third, maltwort or beer; fourth, fermented fruit-juices other than wine; and, fifth, sugar-beets."

W. T. Brannt, on page 20 of his "Practical Treatise on the Manufacture of Vinegar and Acetates," says:

"For consumption on a large scale, especially where only a body of an acid taste is required, the use of so-called 'vinegar essence' (*i.e.*, pure 80 or 90 per cent. acetic acid), prepared from wood, and which, when properly diluted, furnishes ordinary vinegar, will undoubtedly gradually supersede vinegar prepared from alcohol, it being considerably cheaper. And, notwithstanding that the price of vinegar essence is decreasing every year, in regions where wood is plentiful and cheap its manufacture is a well-paying industry, on account of the many valuable by-products (tar, wood-spirit, charcoal) obtained besides acetic acid. Even at the present time for all industrial purposes where acetic acid is required—as, for instance, in the manufacture of tar colours—that obtained from wood is used, and the quantities consumed in the fabrication of table vinegar become larger every year. But the manufacture of vinegar from alcohol and alcoholic fluids will nevertheless continue to flourish, because the product obtained from them actually possesses different properties from the pure acetic acid prepared from wood. Vinegar obtained from pure alcohol, and still more so that from fermented fruit-juices, as wine, cider, skins of pressed grapes, or from malt, contains, besides acetic acid and water, small quantities of bodies which, on account of their being analogous to those occurring in wine, may be designated as 'bouquet-bodies,' and which give to the vinegar an agreeable smell and taste, entirely wanting in acetic acid prepared from wood. These properties are so characteristic that anyone gifted with a sensitive and practised sense of smell can at once distinguish pure acetic-acid vinegar from that prepared from wine, cider, beer, etc. By the addition of volatile oils or compound ethers an agreeable odour can, of course, be imparted to vinegar obtained by diluting pure wood acetic acid with water, but it is impossible to produce the harmonious bouquet peculiar to vinegar from alcohol or fruit-juices, a similar relation existing here as between wine and so-called artificial wine. The latter can be made so as nearly to approach, as regards taste and smell, genuine wine, but a connoisseur will at once detect the difference."

In "Chemistry applied to the Arts and Manufactures," edited by Charles Vincent, wine vinegar, malt vinegar, fruit vinegar, and pyroligneous acid or wood vinegar, are described in separate articles.

In Spon's "Encyclopædia of the Industrial Arts, Manufactures, and Commercial Products," page 2038, the following passage occurs :

"Vinegar is an acid liquid, described in the British Pharmacopœia as prepared from malt and unmalted grain by acetous fermentation. The acid contained in vinegar is acetic acid, and it usually exists in the proportion of 3 to 6 per cent. . . . Although the official prescription is adhered to by some manufacturers, the use alone of those ingredients is by no means usual ; indeed, malt, in many instances, is not in the present day used at all, but for it are substituted artificial glucose and cane-sugar or molasses. These latter are very largely used, and as they produce, chemically speaking, the same result—i.e., acetic acid obtained by fermentation—there can really be no objection to their use."

The "National Dispensary" of Stillé and Maisch states that—

"Vinegar is a dilute acetic acid, obtained by the acetic fermentation of alcoholic liquids. The materials from which it is prepared influence its colour, and to a considerable extent also its odour and taste."

In Pereira's "Materia Medica" it is stated that—

"The acetic acid of commerce is derived from two sources—vinegar and pyroligneous acid ; the first is procured by exciting the acetous fermentation in certain liquids, the other by the distillation of wood."

A. Wynter Blyth, in "Foods: their Composition and Analysis," describes under "Varieties of Vinegar" the various fermentation-products, and classes pyroligneous acid among the adulterants of vinegar.

A. H. Hassall, in his "Food ; its Adulterations and the Methods for their Detection," published in 1876, gives the following "definition of adulteration" of vinegar :

"Free sulphuric acid beyond the quantity allowed by law, or any other mineral acid, or vegetable acid, excepting acetic acid, derived from the manufacture of the vinegar ; water in such proportion as to reduce the acetic acid to below 3·5 per cent. It is questionable whether it would not be well that the law should be abolished which allows of the addition of one part of sulphuric acid in 1,000 parts of vinegar."*

On page 629 of the same work Hassall states :

"The different kinds of vinegar may, according to their source, be divided and classified as malt, wine, cider, beet, sugar, and wood vinegars ; but occasionally other fruits, as gooseberries and currants, are employed for the manufacture of vinegar."

From this passage, taken alone, it might conceivably be held that Hassall regarded acetic acid from wood as a true variety of vinegar, but the definition already given excludes this interpretation, in addition to which Hassall states, a few pages later (635), that—

"The principal adulterations of vinegar are with water, sulphuric acid, burnt sugar, and sometimes with acid substances, as chillies and grains of paradise, and also with acetic and pyroligneous acids."

On the following page (636) he enters into greater detail, thus :

"Very commonly, after the manufacture of vinegar has been completed, the strength is brought up by an addition of acetic acid. We are of opinion that this

* This statement respecting the legality of adding sulphuric acid to vinegar is of very doubtful accuracy. So far as I can ascertain the fact, the addition of 1 gallon of sulphuric acid to 1,000 gallons of vinegar was formerly permitted under an Excise regulation, but the addition being found unnecessary for its intended purpose, the permission was subsequently withdrawn. One gallon per 1,000 was of course very different from 0·1 per cent., the quantity commonly said to have been allowed.

practices is to be regarded as an adulteration. To allow of this addition would be to acknowledge that a mixture of acetic acid and water really constitutes vinegar, which is far from being the case, since genuine vinegar contains extractive matters of different kinds, as well as certain volatile principles, and which (*sic*) affect both the aroma and the flavour."

In the British Pharmacopœia vinegar is defined as "an acid liquid, prepared from a mixture of malted and unmalted grain by the acetous fermentation." The specific gravity is from 1.017 to 1.019, and it is to contain about 5.41 of real acetic acid ($C_2H_4O_2$).

The vinegar of the German Pharmacopœia is required to contain at least 6 per cent. of absolute acetic acid. In Russia the minimum limit of strength is 5 per cent.; in Austria, 6; in Belgium, 5.6; in France, 8 to 9; and in the United States, 4.6 per cent.

In 1874 the Society of Public Analysts adopted 3 per cent. of real acetic acid as the minimum limit of strength for vinegar.* This limit certainly cannot be said to err on the side of too great stringency, and there have been very few prosecutions for the sale of vinegar containing less than this very moderate proportion of acetic acid.

With regard to the sale of vinegar, the pharmacist stands in a far more delicate position than the general dealer, for it might be argued, with some plausibility, that, when purchased of a registered pharmacist, an article recognised in the British Pharmacopœia ought to comply with the description of it given by that authority. This would limit the "vinegar" to be sold by pharmacists to the very best quality of malt vinegar; and if the B.P. definition of vinegar were legally applied to-day, a considerable proportion of the trade would probably be caught tripping.

With the single exception of Brannt, who appears to hold that vinegar may be legitimately manufactured from wood acid, while admitting that the product is inferior to the fermentation acid, all the authorities above quoted agree in regarding true vinegar as an acid liquid produced by the acetous fermentation of alcoholic liquids, and consequently regard acetic acid from wood as not answering to the description of genuine vinegar.

Detection of Cotton-seed-Oil in Lard. F. Gantter. (*Zeitsch. f. anal. Chem.*, xxxii. 303).—The author states that he has met with several samples of cotton-seed-oil which failed to give a silver-reaction, and also a number of samples of lard strongly suspected of adulteration with cotton-oil, which gave no indications by Becchi's test. He concludes that some treatment of the oil is now adopted with a view to circumvent analysts. He has modified the sulphuric acid test, which depends upon the dark coloration produced with cotton-oil, but not with lard, and has in this respect followed Flückiger (*Zeitsch. f. anal. Chem.*, vol. x., p. 235), who recommended dilution of the oil to be tested with ether, benzol, chloroform, or carbon bisulphide, in order to moderate the action of the sulphuric acid. He takes 1 c.c.

* By the term "real acetic acid" there is good reason to believe that acetic anhydride was intended. Three per cent. of $(C_2H_3O)_2O$ corresponds to 358 per cent. of $(C_2H_3O)OH$.—A. H. A.

of the oil or fat to be tested, adds 10 c.c. of petroleum ether, and a single drop of strong sulphuric acid, shaking violently at once. Pure lard becomes straw-coloured, or faint reddish-yellow; after some time the solution becomes clear and colourless, or nearly so, while dark-red droplets separate. Cotton-oil at once becomes dark-brown or black, and the solution remains so for a very long time. Mixtures take on a more or less dark-brown shade; even 1 per cent. of cotton-oil mixed with lard showing the reaction plainly. Olive-oil behaves in a similar manner to lard, but earth-nut-oil resembles cotton-oil.

The author also gives a number of iodine figures for pure and mixed lard, which differ considerably from those found by others. He is of opinion that pure lard should not absorb more than 27 per cent. of iodine. O. H.

NOTE BY THE ABTRACTOR.—The coloration produced by sulphuric acid is almost entirely dependent upon the quantity of sulphuric acid added. A sample of lard, dissolved in petroleum ether, as directed by the author, remained uncoloured by a single small drop of sulphuric acid, with a large drop became yellowish, and brown with two drops. The same changes were observed with a number of other samples. It appears to the abstractor that neither the method nor the iodine figures given by the author are trustworthy.

The Detection of Saccharin in Beer. F. Gantter. (*Zeitsch. f. anal. Chem.*, xxxii. 309.)—Börnstein (*Zeitsch. f. anal. Chem.*, vol. xxvii., p. 167) has recommended, as a most delicate reaction for saccharin, heating the substance to be tested with resorcin and strong sulphuric acid, fluorescein being formed. The author, who examined a number of samples of beer for saccharin, found that the reaction is not conclusive of the presence of saccharin, since beer perfectly free from that body yields a strong fluorescence when the residue from the acid and ether treatment is heated with resorcin and sulphuric acid. He finds that hop-resin and ordinary colophony behave similarly in this respect to saccharin. He relies solely upon the taste of the residue, and operates as follows: 500 c.c. of beer are evaporated to a syrup, precipitated with alcohol, the alcoholic solution acidified with HCl, and then evaporated. The residue is shaken out with ether, the solvent evaporated, and the residue tasted. He is of opinion that the method proposed, which depends upon the detection of sulphur in such residue, is of no use, since the amount of saccharin which is likely to be present in half a litre of beer will be exceedingly minute; consequently but faint indications of sulphuric acid will be obtained after fusion with carbonate and nitrate of potassium. O. H.

On a Method of Titrating Boric Acid. R. T. Thomson. (*Journ. Soc. Chem. Ind.*, xii. 432.)—It is well known that boric acid, when tested in aqueous solution, is neutral to methylorange and slightly acid to phenolphthalein, but as the end reaction is very indistinct, it has not hitherto been found possible to titrate the amount of boric acid alkalimetrically. The author finds that when glycerin is added to a solution of a borate the acidity is increased until, when about 30 per cent. of glycerin, calculated upon the total amount of fluid, is present, the maximum

acidity is reached and the total amount of boric acid is sharply indicated, phenolphthalein being the indicator. The compound NaBO_2 is produced; 1 c.c. of standard soda solution, therefore, corresponds to 0.0620 grammes H_3BO_3 .

When boric acid is to be estimated in borax or in mixtures of borax and boric acid, an aqueous solution is made, methylorange added, and standard acid to neutrality. Thus the total amount of combined alkali is obtained. The whole of the boric acid is now free. Phenolphthalein is now added, and sufficient pure glycerin until the amount of the latter is at least 30 per cent. of the total fluid, and the acidity titrated, 1 c.c. being equal to 0.0505 $\text{Na}_2\text{B}_4\text{O}_7$, or 0.0955 $\text{Na}_2\text{B}_4\text{O}_7 + 10\text{H}_2\text{O}$. In the case of the boric acid of commerce, which generally contains salts of ammonia, 1 gramme may be dissolved in hot water, a slight excess of sodium carbonate added, and the solution boiled down to about half its bulk to expel all the ammonia. Any precipitate which forms is filtered off and the filtrate titrated as described.

Boracite or borate of lime is first dissolved in dilute hydrochloric acid, the solution nearly neutralized with caustic soda, the solution boiled to expel carbonic acid, cooled, exactly neutralized, using methylorange, and the boric acid titrated as directed. If much iron be present, this may be removed by a preliminary treatment with carbonate of sodium, and the oxide of iron, as well as carbonates of calcium and magnesium, precipitated and filtered off.

The author is making experiments as to the applicability of the method for the determination of boric acid in milk and other food materials. This new method appears to furnish a satisfactory solution of an important and difficult problem.

O. H.

REVIEW.

TRAITÉ PRATIQUE D'ANALYSE CHIMIQUE ET DE RECHERCHES TOXICOLOGIQUES, par G. GUÉRIN. London: Ballière, Tindall and Cox.

This compendious work covers 470 pages, exclusive of the index, and contains much information on qualitative analysis not usually found within the same covers. The book is typical of the faults and merits of French works on chemistry. Formulæ and equations are conspicuous by their absence throughout the greater part of the work, and in many cases where they might have been employed to great advantage, but graphic formulæ are given of pilocarpine and certain other of the alkaloids. Alkaloids, by the way, are grouped together with the glucosides and other non-basic bitter principles, without a clear distinction being drawn between them. The ptomaines and leucomaines are duly described.

In the portion of the work devoted to inorganic compounds, the ordinary reactions of the metals and acids are fully described, and the rarer metals are afforded greater prominence than is usual in English text-books. The almost entire absence of formulæ in this part of the work leaves the student in ignorance of the exact composition of the precipitates he produces, and seriously detracts from the value of the book. The information given is often tantalizingly incomplete, and occasionally incorrect. Thus, on page 59, the reader is correctly told that lead chromate is soluble in potash, but insoluble in acetic acid, and that barium chromate is soluble in hydro-

chloric and nitric acids; but the further valuable information that the former precipitate is soluble in nitric acid and the latter insoluble in acetic acid is not given. On page 61 the characteristic reactions of cuprous salts with iodides and thiocyanates are not referred to. Schlagdenhauffen's reaction of magnesium salts with sodium hypoiodite (prepared by dissolving, immediately before use, powdered iodine in a 2 per cent. solution of caustic soda until the liquid is coloured strongly yellow) is not well known in England. The reagent gives a voluminous brown-red precipitate with magnesium, or in a very dilute solution a reddish coloration, while lime, baryta and strontia are unaffected. The most important practical reaction of titanium, namely, that of forming a solution in sulphuric acid, or acid sulphates of alkali metals, from which metatitanic acid is precipitated on dilution and prolonged boiling, is entirely omitted. Under zinc, no reference is made to the delicate reaction of hot ammoniacal solutions with potassium ferrocyanide. In describing the reaction of acetates with ferric chloride, the red liquid is said to yield a brown precipitate of ferric hydrate on boiling. No attention is given to the quantitative possibilities of the various reactions described, and the methods for separating the metals and salt radicals are dismissed very summarily, as are the means of dealing with insoluble substances. Methods for qualitatively analyzing gases are given, and there is a chapter on spectrum-analysis, in which the micro-spectroscope is not described. This is followed by methods of toxicological analysis, in which the usual processes for detecting arsenic, phosphorus, hydrocyanic acid, chloral and chloroform are detailed. The isolation of vegetable alkaloids is next described, and the inevitable tables of Dragendorff reproduced. The work closes with a description of "methods and practical processes for the analysis of some substances necessitating more special treatment." The substances considered in this section are limited to water, clay, steel and iron. On page 402 we are told that in 1847, in England, M. Clark (*sic*) indicated a method of rapidly examining waters, and that this method has been perfected in France by MM. Boutron and Boudet, who have written of it under the name of "hydrotimetry." This is simply Clark's soap test, the only apparent innovation being the use of the instrument long obsolete in English laboratories, formerly known as a Binks' burette. A full description is given of the methods of effecting a bacteriological analysis of water.

M. Guérin's work appears to be very unequally written. The whole of the information relating to the commoner metals and acids is given more fully and accurately by Fresenius, but in the case of the less frequently occurring metals and salt-radicals M. Guérin has recorded much useful information. Of quantitative processes he seems to have a wholesome horror, and even when, as in the determination of the saline matters in water, he is compelled to weigh such compounds as barium sulphate, magnesium pyrophosphate and potassium chloroplatinate, he gives no formulæ for these products, and carefully abstains from indicating the method of deducing from the weights obtained the corresponding amounts of the substances sought to be determined.

The book contains a good deal of useful information, and the faults and omissions which detract from its value as a handbook for students, for whom it is professedly written, will not be felt by the skilled chemist, who, on the other hand, will learn how little the work of English analysts is known or appreciated in France. Many of the illustrations will be welcomed as old friends. Excellent coloured diagrams of borax beads and metallic spectra are given.

A. H. A.

CORRESPONDENCE.

To the Editors of THE ANALYST.

SIRS,—I send you the following, in the hope that it may be of interest to your readers, and possibly of service if any of them should be similarly situated.

On the 5th day of May, 1879, I was appointed Public Analyst for the Borough of Neath, as per following resolution, a copy of which was forwarded to me by the late Town Clerk :

“Resolved, that Dr. William Morgan, Public Analyst of Swansea, be appointed Public Analyst for this borough, on the terms mentioned in his letter of the 16th April ult.”

The terms were 10 guineas per annum and 15s. per sample. In due course the sanction of the Local Government Board was given, as provided under section 10 of the Food and Drugs Act, 1875.

All went on well until December, 1892, when a resolution was passed, at a meeting of the Neath Town Council :

“That Dr. Morgan be given three months’ notice, as from the next quarterly meeting of this Council, to terminate his engagement as Public Analyst for this Borough.”

The purport of the above was to terminate my appointment in May, 1893.

The above resolution was forwarded to me by the Town Clerk, to whom I replied as follows :

“I have duly received your letter containing copy of resolution of the Town Council of the borough of Neath, purporting to terminate my appointment as Public Analyst three months from the first Monday in February next. I must, however, respectfully decline to accept such notice until my removal has been sanctioned by the Local Government Board.”

The above letter was read at the January Council meeting, and the following resolution was passed :

“That the Town Clerk do inform the Local Government Board that the appointment of Dr. Morgan as Public Analyst for this borough has been cancelled.”

The following is the reply of the Local Government Board :

“I am directed to acknowledge the receipt of your letter of the 3rd inst., embodying a copy of resolution passed by the Town Council respecting their proposal to cancel the appointment of Dr. Morgan as Analyst. In reply, I am directed to point out that the appointment of an Analyst, under section 10 of the Sale of Food and Drugs Act, 1875, cannot be determined by a Local Authority without the approval of the Board. Before further considering the subject, the Board request to be fully informed of the reasons of the Town Council for desiring to dispense with Dr. Morgan’s services.”

At a Council meeting held on the 6th day of February, 1893, the foregoing letter was read, and it was resolved to reply to it as follows :

" 1st. We have an Analyst who can be seen at any time by the officers of the Corporation.

" 2nd. There is a greater possibility of the townspeople taking advantage of the services of an Analyst who lives in the town.

" 3rd. That as local autonomy is the order of the day, the Corporation are justified in terminating the engagement of their officers at pleasure, and are certainly entitled to prefer the employment of a townsman to that of a stranger."

The following reply, dated March 6th, was sent by the Local Government Board to the Town Clerk :

" I am directed by the Local Government Board to acknowledge the receipt of your letter of the 9th ult., and of its enclosure, with reference to the proposal of the Town Council of the borough of Neath to determine the appointment of Dr. Morgan as Analyst for the borough.

" In reply, the Board direct me to state that there do not appear to them to be any sufficient grounds to justify them in approving of Dr. Morgan's removal from office, and that they are therefore unable to accede to the application of the Town Council."

The above letter was read at the Council meeting held on the 12th April last, and it was resolved to send the following to the Local Government Board :

" That the Local Government Board be informed that Dr. Morgan was appointed under section 11 of the Food and Drugs Act, 1875, and not under section 10; that no confirmation of his appointment was necessary; and that the information conveyed to them as to determining the appointment was merely an act of courtesy on the part of the Town Council."

The following is the reply of the Local Government Board, and read at the meeting held May 1st :

" I am directed to state that the Board have referred to the correspondence, and to the copy of the resolution passed by the Town Council with regard to the appointment of Dr. Morgan, and that they see no ground for the contention that the appointment was not made under section 10 of the Sale of Food and Drugs Act, 1875."

The last resolution of the Council is, " That a copy of the whole of the correspondence be sent to one of the County Members, in order that he may question the President of the Local Government Board in the House of Commons."

I may state that the sole object of the Council was to remove me in order to give the appointment to a young man residing at Neath, and whose father and brother are aldermen of the borough.

I am, yours faithfully,

WILLIAM MORGAN, Ph.D.

Swansea, May 3rd, 1893.

THE ANALYST.

AUGUST, 1893.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

SUMMER MEETING.

THE annual summer meeting of the Society of Public Analysts was held on July 18th at the College of Surgeons, Dublin. Sir Charles Cameron, the President, occupied the chair. Among those present were Professor Tichborne, Dr. Lapper, Mr. A. H. Allen, Mr. M. A. Adams, Mr. Otto Hehner, Mr. A. Smetham, Dr. F. L. Teed, Mr. James Baynes, Mr. Wallis Jenkins, Dr. S. Roche, Mr. Rowland Williams, Mr. MacAllan, Mr. Francis, Dr. Bernard Dyer, and a number of ladies.

The President, in opening the proceedings, said that his first duty was on behalf of the local members of the Society to give a cordial welcome to the members from the other side of the Channel. This was not the first time that the summer meeting of the Society had been held in Ireland; it was, in fact, the third occasion. Twelve years ago, when the British Association met in Dublin, there was a meeting of the Association held in that College, and eight years ago also the summer meeting and dinner in connection with the Association was held in that College. This was the third time that they had assembled there.

Mr. Otto Hehner temporarily took the chair whilst Sir Charles Cameron read his paper on "The Results of the Working of the Adulteration Acts in Dublin."

A discussion followed, after which Professor Tichborne read a paper on "Some Recent Improvements in the Babcock Machine," and Mr. A. H. Allen a paper on "Vinegar: its Nature and Manufacture."

The following papers were taken as read: "Notes on Butter," by Mr. Otto Hehner; "A General Method for the Analyses of Oils and Fats," by Mr. H. Droop Richmond; "On the Detection of Heated Cotton-Seed Oil in Lard," by Mr. W. G. Crook; "Note on the Estimation of Chlorine in Certain Waters," by Mr. T. Fairley; "Some Simple Appliances for Measuring the Consistency of Semi-solids, with Note on a New Method of Butter Examination," by Mr. C. E. Sohn.

A vote of thanks to Sir Charles Cameron for presiding terminated the proceedings.

In the evening a banquet took place in the Shelbourne Hotel. Sir Charles Cameron presided. A special and attractive feature of the occasion was the presence of ladies.

Amongst those present were the Lord Mayor and Lady Mayoress, the Right Hon. Alderman Meade and Mrs. Meade, Sir Andrew Reed, C.B., Sir William Kaye, C.B., Sir Philip Smyly, Dr. Meldon, D.L., and Mrs. Meldon, Dr. and Mrs. Oulton.

Dr. and Mrs. Heuston, Mr. and Mrs. Otto Hehner, Mr. Allen, Dr. and Mrs. Charles Poll, Dr. Carte, Dr. and Mrs. Broomfield, Dr. and Mrs. Jacob, Dr. and Mrs. Teed, Dr. Arthur Benson, Dr. and Mrs. Harley, Dr. Magrath, Dr. and Mrs. Tichborne, Alderman Maguire, Rev. B. Gibson, Professor and Mrs. Lapper, Dr. and Mrs. Power O'Donoghue, Mr. Williams, Mr. Smith, Mr. Smetham, Mr. Jenkins, Mr. Baynes, Dr. and Miss Adams, Mr. and Mrs. A. O'Kennedy, Mr. J. MacAllan, Mr. Francis, Professor Roche, Mr. O'Meara, T.C., Dr. and Mrs. Alexander Smith, the Rev. Canon Bagot, Dr. Dyer, Dr. and Mrs. Knott, Mr. and Mrs. Farrell, Dr. Ormsby, Mr. Melfort D'Alton, Dr. and Mrs. Smyly, Mr. Coffey, Dr. Byrne, Mr. E. Douglas Cameron, Mr. E. Stewart Cameron, etc.

After dinner, the President proposed "The health of her Majesty the Queen and the Royal Family."

The toast was received with applause.

The President next proposed "The Corporation of Dublin." He said that at no previous period of its history did the Corporation of Dublin perform a more useful work to the citizens of Dublin than at present. In old times the chief business of the Corporation was to amuse themselves and spend the revenues of the city very much to their own advantage and to that of their friends; but in the present day the Corporation of Dublin was an eminently representative body—as, in fact, were most corporations in the United Kingdom—and they administered the affairs of this city with every desire to afford the greatest possible benefit to the citizens at large. The President, having spoken of his long connection as City Analyst with the Corporation of Dublin, proposed the health of that body, and coupled with the toast the name of the Lord Mayor.

The toast was drunk with applause.

The Lord Mayor, in responding, said that about fourteen or fifteen years ago they saddled themselves with a public analyst, and in doing so they had taken on their backs a veritable old man of the sea, for since that time peace and quietness and contentment had left the precincts of the Corporation. Acts of Parliament had been passed in abundance, and new taxes had been imposed on the city, and despite all that they had done fresh sacrifices were called for every year, and when Sir Charles Cameron laid down the law they had only to carry out his will, whatever it was, and it was a moot point whether it was wicked or benevolent (laughter). Science was said to have achieved great things, but the thing had gone far enough, and they should think of giving the citizens a rest, and not be too ready to increase the taxation of the city. Those, however, who remembered what the city was fourteen or fifteen years ago should recognise the progress and improvement made in this city, which in many respects supplied models and examples for the cities of the United Kingdom. Irishmen appeared to have been born for the express purpose of showing Englishmen and Scotchmen the way they ought to go (laughter). They all knew what a high opinion was entertained of Irishmen at the other side of the Channel; for not only were they going to allow Irishmen to manage their own affairs, but they desired to retain them at Westminster to see that English and Scotch affairs might be properly looked after (laughter).

The toast having been honoured, and an excellent vocal and instrumental duet having been given by Dr. and Mrs. Power O'Donoghue,

The Lord Mayor proposed "The Society of Public Analysts of Great Britain and Ireland," and coupled with it the name of Sir Charles Cameron, of whose services to the city as Public Analyst he spoke in commendation.

Sir Charles Cameron, in responding, said he felt flattered by the observations made by the head of the municipality in reference to the services which he had rendered to this city, of which he was proud to be a native born. It was a matter of pride to him that he had been elected President of the Society of Public Analysts, which included in its ranks the larger number of the most distinguished chemists in these countries. Its members were to be found all over the United Kingdom, in India, and in the Colonies. It was often said that if they wanted to know what was the opinion entertained of a man they should look for that opinion to the members of his own profession, and as an Irishman he was proud they had elected him as its president. He trusted that the members of the Society appreciated the reception accorded to them on the present occasion, and would be induced again to visit Dublin, a city which invariably accorded a friendly reception to the British Association and other distinguished bodies.

Sir Andrew Reed, C.B., in a happy and eloquent speech, proposed the toast of "The Ladies," which was duly honoured.

The Rev. Dr. Gibson and Dr. Smyly responded.

Alderman Meade, LL.D., proposed "The Health of the Officers and Council of the Society of Public Analysts," and coupled with the toast the names of Dr. Adams, Mr. Allen, Mr. Hehner, and Dr. Dyer.

The toast was honoured and responded to, and, with the toast of "The Press," the proceedings were brought to a close.

Dr. Smyly and other gentlemen made contributions to the pleasure of the evening by vocal and instrumental music.

NOTES ON (a) RICE-OIL; (b) MAIZE-OIL.

By ALFRED SMETHAM, F.I.C.

(Read at the Meeting, June 7th, 1893.)

SOME time ago a sample of rice-oil, obtained from Rangoon rice meal by means of hydraulic pressure in a clean press, came into my possession. The oil had a somewhat dirty-greenish colour, was semi-solid at the ordinary temperature, with white crystals giving it the appearance of the "figging" of soft-soap.

On examination, the oil, after filtering, yielded the following results:—

Iodine absorption	96.4%
KHO required for saponification	19.32%
= Saponification equivalent	290
Free fatty acids (KHO \times 5)	64.0%

The proportion of free fatty acids in this oil being so high, I thought it desirable to ascertain whether the result was an exceptional one, or whether it fairly represented the character of the average rice meal imported. I therefore extracted, by means of ether, the oil from a number of samples of rice meal, which had been sent to me for analysis by different people, and after evaporating off the ether, the mixed oil was filtered through a dry filter in the water-oven at a temperature of 100° C.

On examination this oil yielded the following results:—

Iodine absorption	99.9%
KHO required for saponification	19.20%
= Saponification equivalent	292
Melting-point	29° C.
Free fatty acids (KHO × 5)	77.20%

As this mixed oil showed a higher percentage of free fatty acids than the original pressed oil, I obtained a sample of recently-imported Rangoon rice, and separated, by hand picking, the husk from the clean seed. On analysis these gave:—

	Cleaned Rice.	Husk (Shudes).
Moisture (loss at 100° C)	12.65	9.70
Oil	2.20	1.16
Albuminoids (N × 6.25)	7.75	3.25
Starch and digestible carbohydrates	75.34	32.44
Cellulose	.66	39.90
*Ash	1.40	13.55
	<hr/> 100.00	<hr/> 100.00
* Including SiO ₂	10	13.20

The free fatty acids in the oils extracted were:—

	Cleaned Rice.	Husk.
Free fatty acids	31.60%	34.00%

I then obtained what I believe to be the rice meal obtained from the rice I had previously examined, and analyzed it within a day or so of the time it was manufactured. I may here state that what is known as rice meal is the portion obtained by dressing the rice under emery runners, and does not, therefore, include the interior, or starchy portion of the seed.

This meal gave on analysis the following figures:—

Moisture (lost at 100° C.)	...	11.20
Oil	...	8.16
Albuminoids (N × 6.25)	...	12.50
Starch and digestible carbohydrates	...	57.83
Cellulose	...	4.46
*Ash	...	5.85
		<hr/> 100.00

* Including silica, 2.00.

From this meal a considerable quantity of oil was extracted in the manner before described, and on examination it yielded the following results :—

Iodine absorption	96.9%
KHO required for saponification	19.58%
= Saponification equivalent	287
Free fatty acids (KHO \times 5)	43.40

From these results it would be difficult to say whether the oil originally formed in the seed was a neutral one; but whether this is so or not, it is quite evident that the oil of rice meal, whether produced in England or imported as meal, contains a large percentage of free fatty acids. The percentage of free acid appears to be greater in the oil of the imported meal than that extracted from the seed by me, but whether this is due to the fact that decomposition takes place more rapidly in the meal than in the seed, or whether it is due to the fact that at Rangoon the rice is not dressed so perfectly as in England, I am unable to say; but I am strongly of opinion that it is chiefly brought about by differences in the processes of manufacture. The English meal, as a rule, contains from 8% to 9% of oil, while the Rangoon averages about 15%.

The following figures, obtained from four different samples, are in accordance with the preceding :—

	Rangoon Meal.	American.	Rangoon, (New Season).	
			1st Sample.	2nd Sample.
Percentage of oil in meal	15.46	11.03	15.86	15.36
Free fatty acids in oil	76.2%	52%	67.6%	50.6%

Maize-Oil.—At the latter end of last year an oil produced in the United States from maize was being offered to the soap-makers of this country. It was a bright, clear, yellowish oil, sweet to the taste and smell, and was offered at a price somewhat above cotton-seed oil. It was, however, sufficiently cheap to render it available as an adulterant of the dearer oils. The following figures may, therefore, prove of interest :—

Iodine absorption	116.3%
KHO required for saponification	19.88%
= Saponification equivalent	282

ON RAPID SAPONIFICATION FOR ANALYTICAL PURPOSES.

By ALFRED SMETHAM, F.I.C.

SOME time ago, while examining some mixed oils by the Koettstorfer process, I was struck with the difficulty experienced in completely saponifying the whole of the vegetable oil present, and, in the presence of mineral oil, of ascertaining exactly when the reaction was complete. This led me to consider that the difficulty was caused by the insolubility of the oil in the alcohol, and the consequently limited surface exposed by it to the action of the potash; and that, if that were so, any menstruum in which the oil and alkali were both soluble would allow of very rapid saponification. Although I have not been able to devise such perfect conditions as these just formulated, I have, by the very simple method of adding a little ether to the fat

previous to the addition of the alcoholic potash arrived at practically the same result. The ether seems to act as a carrier, and saponification takes place very rapidly. The addition of ether also has the further advantage that in the presence of comparatively small quantities of mineral oil, not only is the animal or vegetable oil rapidly saponified, but a clear solution of the mineral oil is obtained, thus satisfying the operator that the mineral oil does not enclose any saponifiable matters—a point of no small satisfaction in the case of mixed oils.

Finding that in practice a considerable advantage was obtained by the addition of ether, I made a series of experiments to ascertain what conditions were the most favourable, and to what extent the process might be relied upon.

The saponification was conducted in flasks of about six ounces capacity, with glass tubes about three feet long, fitted by means of corks. After the addition of the alkali to the fat, the flask was placed upon a hole in a water-bath in which water was kept briskly boiling, and the flasks and contents were shaken at frequent intervals. The conditions of temperature, etc., were as nearly the same in all the experiments as possible. The alcoholic potash was approximately semi-normal.

The following are the results obtained, the percentages being calculated, for the sake of comparison, on the weight of saponifiable fat taken :

Tallow :

Fat taken.				Reagents used.	Time occupied. Minutes.	KHO consumed.
(1)	3 grms.	30 c.c. KHO sol.	117	19.50%
(2)	3 grms.	{ 30 c.c. KHO sol. + 10 c.c. ether }	66	19.61%
(3)	3 grms.	{ 30 c.c. KHO sol. + 20 c.c. ether }	11	19.39%
(4)	3 grms.	{ 30 c.c. KHO sol. + 30 c.c. ether }	10	19.20%
(5)	3 grms.	{ 30 c.c. KHO sol. + 40 c.c. ether }	9	16.72%
(6)	2.5 grms. tallow 540 kerosene }	{ 30 c.c. KHO sol. + 20 c.c. ether }	25	19.56%
(7)	2.5 grms. tallow 5 grms. paraffin wax }	{ 30 c.c. KHO sol. + 20 c.c. ether }	50	19.52%
(8)	2.5 grms. tallow 5 grms. paraffin wax }	{ 30 c.c. KHO sol. + 50 c.c. ether }	16	19.12%

Colza-oil :

(9)	3.241 grms.	30 c.c. KHO sol.	122	17.68%
(10)	3.545 grms.	{ 30 c.c. KHO sol. + 20 c.c. ether }	33	17.56%
(11)	3.676 grms.	{ 30 c.c. KHO sol. + 40 c.c. ether }	8	16.01%

Olive-oil :

(12)	3.000 grms.	30 c.c. KHO sol.	102	19.20%
(13)	3.134 grms.	{ 30 c.c. KHO sol. + 20 c.c. ether }	15	19.86%
(14)	3.015 grms.	{ 30 c.c. KHO sol. + 40 c.c. ether }	15	18.92%

Coprah-oil:

Fat taken.				Reagents used.	Time occupied: Minutes.	KHO consumed.
(15)	3 grms.	35 c.c. KHO sol.	35	25.88%
(16)	3 grms.	{ 35 c.c. KHO sol. + 20 c.c. ether }	15	25.92%
(17)	3 grms.	{ 35 c.c. KHO sol. + 40 c.c. ether }	15	25.94%

Raw Linseed-oil:

(18)	3.028 grms.	30 c.c. KHO sol.	20	18.96%
(19)	3.023 grms.	{ 30 c.c. KHO sol. + 20 c.c. ether }	5	19.12%
(20)	3.027 grms.	{ 30 c.c. KHO sol. + 40 c.c. ether }	15	18.97%
(21)	3.003 grms. linseed 6 mineral lubricating oil	}	...	{ 30 c.c. KHO sol. + 20 c.c. ether }	5	15.52%
(22)	3.019 linseed 521 mineral lubricating oil			{ 30 c.c. KHO sol. + 20 c.c. ether }	15	19.00%

Refined Cottonseed-oil:

(23)	2.947 grms.	30 c.c. KHO sol.	60	19.48%
(24)	3.148 grms.	{ 30 c.c. KHO sol. + 20 c.c. ether }	15	19.52%
(25)	3.221 grms.	{ 30 c.c. KHO sol. + 40 c.c. ether }	15	19.52%

In criticising the foregoing results, it should be kept in mind that in nearly all those instances in which the KHO consumed deviates from the correct amount to any appreciable extent, the test was purposely terminated in order to ascertain the least possible time in which saponification was complete, and these low results must not, therefore, be used to cast any reflection on the accuracy of the process.

The first test with each oil, in which only alcoholic potash was used, was continued until no more particles of oily matter could be detected with the eye, at which point saponification was judged to be complete. In no case, whether with or without ether, was any titration made while any oil was perceptible; and it will, therefore, be seen that not only must solution take place, but that time must be given to complete the reaction.

In experiment 7 the boiling was continued, to see whether the paraffin wax dissolved; but as the bulk of the ether boils off in the first fifteen or twenty minutes, no such solution took place.

In experiment 13 the liquid became clear in three minutes, in No. 22 in four minutes, and in 24 in seven minutes; but the digestion was continued in each instance for a quarter of an hour.

Judging from experience during the progress of these tests, it seems quite clear that no sufficient advantage is gained by increasing the quantity of ether beyond 20 c.c. In fact, a larger quantity seems to have a detrimental effect by lowering the boiling-point of the mixture. If, as I imagine, the ether simply acts as a carrier of the oil to the KHO, the longer time necessary for saponification in the presence of a larger quantity of ether is quite intelligible.

That heat is not necessary for complete saponification was shown by the fact

that, in the presence of 40 c.c. ether, 3 grammes of tallow was entirely decomposed on standing in the laboratory over night at the ordinary temperature.

In practice it would be a safe precaution to allow the greater part of the ether to evaporate before titration—although there is no need to do so if time be given for the completion of the reaction. The time occupied for 3 grammes of fat and 20 c.c. ether should in no instance be less than fifteen minutes; but if even double that time be allowed, a very marked advantage over the process as ordinarily worked will be obtained.

DISCUSSION.

Mr. Alfred H. Allen congratulated Mr. Smetham on having initiated a number of very interesting experiments, and on arriving at results well worthy of the careful consideration of members of the society. Respecting the results obtained from rice-oil, it was a very curious but not sufficiently-explained fact that cereal oils often seemed to consist largely of free fatty acids. It was, at any rate, true of oat-oil, within his own experience, and it was now shown to be the case with rice-oil. He was very glad to see that the percentage of potash required for saponification was given. He protested against German chemists calling the quantity of potash required per thousand parts of oil the "saponification-equivalent." They were confusing terms, and when abstractors in this country translated foreign papers into English, the term "saponification-equivalent" was misapplied to such figures. There was only one correct way of using the term "saponification-equivalent," which should always signify the number of parts of the fatty material saponified by 56.1 parts of KHO or 40 parts of NaHO . With respect to the use of ether in the saponification experiments, it was disappointing to find that the author could not saponify completely in the presence of the larger amounts of ether. The figures he obtained were, of course, what might be expected. Anything which would shorten the labours of analysts, without affecting the accuracy of results, was to be welcomed, and he thought the society ought to feel indebted to Mr. Smetham for pointing out this method of securing a more perfect mixture, and therefore a more rapid saponification; but he could not help thinking that in a great many cases it was not worth while to make the addition, and if Mr. Smetham would only take the trouble to agitate the caustic potash with the fat, he would find a great increase in the rapidity of saponification. His (Mr. Allen's) experience was that fifteen minutes was amply sufficient to effect saponification in ordinary cases.

Mr. H. Droop Richmond asked Dr. Dyer if there was anything in the paper which gave a clue to the strength of the alcohol used. It would seem that the alcohol used must have been extremely weak if two hours were required for saponification. He had frequently performed the experiment in ten or fifteen minutes by heating in a flask attached to an upright condenser.

Mr. Blount felt convinced that it was a question of the strength of the alcohol used. He had frequently had occasion to saponify rape-oil, and he was sure that the saponification was quite complete in twenty minutes over the water-bath. He had used absolute alcohol, and frequently shook the flask.

Mr. Otto Hehner said that in his experience when the alcohol was sufficiently

light for the oil to sink in it, it was strong enough; but when the fat floated, then the experiment had better be recommenced. He believed that if Mr. Smetham had boiled the solution vigorously under a condenser the saponification would have been much more rapid. The most difficult body to saponify—ordinary wax—was completely saponified in an hour, provided the ebullition was violent. There seemed to be some advantage in using ether, but it had the disadvantage of lowering the temperature of the mixture. If a fatty body were saponified on the water-bath, then it appeared advisable to add a little ether. The figures of the other experiments which Mr. Smetham had brought forward under the headings of rice- and maize- oil were very valuable, and pointed out a very useful field of investigation, as maize-oil was now brought into commerce. He hoped that Mr. Smetham would tell them in a future paper something further about this substance. It was certainly a very interesting fact that these oils had so high an acidity. From what was known of the formation of oils, he was convinced that the oil, as formed in the plant or ultimately deposited in the animal, was always neutral. There were exceedingly few, if any, exceptions to this. One, which was considered an exception for many years—cod-liver-oil—had been shown not to be so. Some cod-liver-oils contained a very large percentage of acid, and a small percentage of fatty acids had always been found even in the freshest oil; but if the liver were taken out of the fish as soon as possible after it was killed, and the oil was pressed out at once, it was free from acidity; but if, on the other hand, the oil was allowed to remain in the liver for a day, it became acid. The acidity of cod-liver-oil was entirely due to the fishermen collecting livers some distance from the shore. As a considerable period elapsed before the seed to which Mr. Smetham had referred reached this country, naturally the amount of oxidation and decomposition in the seed was considerable.

GINGER: WITH SPECIAL REFERENCE TO DISCRIMINATION BETWEEN GENUINE AND "EXHAUSTED" SPECIMENS.

By BERNARD DYER, D.Sc., and J. F. H. GILBARD.

(Read at the Meeting, June 7th, 1893.)

It is well known that ginger which has been "exhausted" or "extracted" by mineral water manufacturers finds a ready market as an adulterant of genuine ground ginger, and that the detection of such adulteration is infrequent, although dealers in honest spices have long groaned under the unfair competition to which such adulteration subjects them. The word adulteration is used advisedly, because the case is precisely analogous to the admixture of skim milk with whole milk.

The chemistry of ginger has been laboriously worked out by Dr. Thresh, whose classical papers on the subject may be found in the back volumes of the *Pharmaceutical Journal*. In THE ANALYST only two papers have appeared on the subject of ginger, the first by Mr. E. W. T. Jones, in 1875, on "The Amount of Starch in Ground Ginger," the second by Mr. W. C. Young, in 1884, on "Some Analyses of Ginger." Mr. Young's opening paragraph runs thus:

"These analyses were made in the hope that some data would be found which would enable analysts to distinguish between genuine ground ginger and that to which exhausted ginger had been added. As will be seen by the results, my hope was not realized, the constitution of the samples being so widely different."

The points determined by Mr. Young were moisture, ash, ash soluble in water, "aqueous extract," "mucilage," "alcoholic extract," "resin" and cellulose. The method adopted for the determination of alcoholic extract was an arbitrary one, and the extraction may or may not have been complete. How "resin" was determined the author does not state. He does not seem to have realized the full value of the determination of *soluble ash*, which is, as we shall show, an item of valuable significance. Perhaps Mr. Young had not made sufficient comparative trials with exhausted ginger, or we cannot think he would have so despairingly dismissed the subject.

The question was some time back referred to one of us by Messrs. Joseph Travers and Sons, the well-known spice merchants and grinders, who were good enough to supply him with some typical specimens of genuine commercial ginger, and through whose help he was also able to obtain a number of samples of "exhausted ginger" (varieties unknown), *i.e.*, of ginger which had been utilized by makers of aerated waters.

The constituents, or groups of constituents, which it appeared to us desirable to determine were :

Moisture (loss at 100° C.)
Volatile essential oil.
Fixed ethereal extract.
Alcoholic extraction after ether extraction
Ash.
Ash soluble in water.

There is no satisfactory method of determining the volatile essential oil, although it is one of the most important constituents of ginger.

Dr. Thresh determines it by "drying" the ether extract at a low temperature and subsequently at a higher temperature, until it ceases to lose weight. The difference between the preliminary and the final "drying" he takes as volatile essential oil.

This is obviously a clumsy and unsatisfactory method, and we believed at one time that we had hit upon a better and a fairly quick and ready method of making, if not an accurate, at all events a comparative, determination of volatile oil. Our proposed method was to distil a small quantity (one to two grammes) of the powdered ginger with successively-added small quantities of water, collecting the distillates all together in one bottle. When the "oil" is all over, the contents of the bottle, well shaken up, exhibit a turbidity, and we hoped that the degree of turbidity would approximately measure the volatile oil. The first experiments made were promising enough and showed overwhelming differences in turbidity between the distillates from a genuine and from an exhausted sample; but in the further prosecution of our experiments the method broke down, for we found that, as perhaps might readily have been anticipated, the steam carried over not merely volatile oil, but fatty and resinous constituents of the ginger, so that the turbidity of the distillate was no real

measure of the amount of essential oil present. We therefore fell back on the simple, though somewhat lame, method of Dr. Thresh, merely placing the flasks containing the ethereal extract on a shelf above the water-oven until they lost only a few milligrammes weight per hour, and drying to final constancy in the water-oven itself.

Five grammes of the powdered ginger were taken for ethereal extract, the extraction being performed in a Soxhlet's apparatus. The ether extraction is often scarcely completed in one day, so it is desirable, after a few hours, to remove the flask with the first portion of the extract, and proceed to dry it as above described; then to substitute a second flask and continue the extraction for a second day, adding the residue thus obtained to the first residue.

Absolute alcohol was then substituted for the ether in the Soxhlet apparatus, and the alcoholic extract determined. Complete alcoholic extraction generally requires two days.

The ash was determined in five grammes and washed with hot water. The insoluble matter was weighed, digested in acid, and the siliceous matter determined.

The following are our results :

Unexhausted Gingers.	Moisture. Loss at 100° C.	(Approximate) volatile essential oil.	Fixed ethereal extract.	Alcoholic extract after ether extraction.	Total ash, less sand.	Ash soluble in water.	Ash insoluble in water, less sand.
Best Jamaica	13.6	0.7	3.0	3.1	3.1	2.4	0.7
Jamaica	13.4	1.2	3.9	3.8	3.9	3.0	0.9
Cochin	12.9	1.2	4.7	3.1	3.6	2.3	1.3
"Original" African	14.4	2.9	6.4	2.7	4.0	2.4	0.6
Washed African	14.1	2.5	7.0	2.1	4.1	1.9	2.2
"Original" Bengal	14.6	1.5	4.5	2.1	3.9	2.6	1.3
Exhausted Gingers. (Varieties unknown.)							
A	12.1	0.8	5.2	1.2	2.1	0.4	1.7
B	11.8	0.5	3.0	1.2	1.2	0.3	0.9
C	11.8	0.4	4.7	1.4	2.3	0.4	1.9
D	11.7	0.9	4.9	1.5	2.2	0.5	1.7
E	11.9	0.5	3.0	0.8	1.1	0.2	0.9
F	11.5	0.7	4.1	1.1	1.9	0.3	1.6

The percentage of siliceous matter varied from 0.2 to 1.5 in the genuine samples, and from 0.3 to 0.4 in the exhausted ones.

The "volatile oil" is obviously too variable to be taken as a basis for the quantitative estimation of admixture in adulterated samples, though it may, in conjunction with other features, have some significance. The "fixed ethereal extract" has little if any value, owing to its variability in genuine ginger.

The extract obtained by alcohol after complete removal of the ethereal extract, appears, however, to be a valuable item to the analyst. In the genuine gingers it ranges from 2.1 to 3.8 per cent., with an average of 2.8 per cent. In the "exhausted samples" it ranges from 0.8 to 1.4 per cent., with an average of 1.2 per cent.

The total ash in the genuine gingers (excluding sand) ranges from 3.1 to 4.1 per

cent. In the "exhausted" samples it ranges from 1.1 to 2.3 per cent., averaging 1.8 per cent.

The *ash soluble in water* ranges in the genuine gingers from 1.9 to 3 per cent., averaging 2.7 per cent. (Mr. Young in seven authenticated samples found the soluble ash to range from 1.8 to 2.6 per cent.).

In the "exhausted" samples the soluble ash ranges from 0.2 to 0.5, averaging 0.35 per cent.

While it appears to be unfortunately true that some samples of genuine ginger may be mixed with a considerable quantity of exhausted ginger without affording conclusive analytical evidence of the fact, it is at the same time, we venture to think, obvious that in a great many cases the addition may be detected, and that at all events much of the flagrant adulteration of ground ginger which is commonly believed to exist might be prevented by analytical vigilance.

DISCUSSION.

Mr. Hehner said that it was important that analysts should be able to deal with an article which was so notoriously adulterated as ginger. To his knowledge a very large bulk of spent ginger which was used for trade purposes was re-mixed and sold to the unwary as whole ginger.

Mr. E. J. Bevan asked the authors of the paper how they accounted for the ash soluble in water, and the total ash being so much reduced. Was it the case that the ginger was first extracted with alcohol? If the ash was determined after the extraction with alcohol, it ought to be considerably higher.

Dr. Dyer replied that it was weak alcohol which dissolved out the ash. Strong alcohol did not diminish the ash. Weak alcohol was used in making extracts, because in that case a finer flavour was obtained, objectionable resins being excluded.

Mr. Budden asked Dr. Dyer if he knew that it was a fact that in the trade a great number of different methods of extraction were employed. In the case of ginger wine made from raisin wine, etc., the ginger was bought in the condition of root, roughly crushed and then "steeped," as it was termed in the trade, for a long time in a liquor containing 25 per cent. of proof spirit. Only the more delicate flavoured matter was abstracted by that means, and the extracted or "spent" ginger was then sold for the purpose of adulterating ginger and spices, and a certain proportion of it found its way to the lower classes of gingerbeer makers. It had a distinct value as a flavouring material. Probably the whole of the volatile oil had been extracted, but all the pungent resinous matters were left behind. He further wished to ask whether the authors of the paper had made any experiments on the solubility in pure water, without the addition of spirit. He believed he was right in stating that the most valued flavouring matters of ginger were really capable of being extracted by water, and that the method which was adopted by the manufacturing chemist, who specially prepared ginger for the manufacture of aerated beverages, was first of all to extract the ginger fully with very strong alcohol, and subsequently to precipitate the whole of the resinous matter. After evaporation in *vacuo*, a residuum was obtained which was practically soluble in water, without any

further precipitation of resinous matters. In support of the view that the real flavouring principle of the ginger root was soluble in water, it might also be stated that occasionally ginger comes into the market which has been deprived of a small percentage only of its flavour by boiling with water. Subsequent drying leaves this product very much in the condition of an inferior genuine ginger, but if it be not sold as "spent ginger" the fraud is obvious.

Mr. Sidney Harvey said that it was very important to manufacturers of some of the many aerated ginger-beers prepared, not by fermentation, but by charging with carbonic acid gas a solution of sugar, and acid, and ginger extract, to avoid anything like turbidity, and the first aim of makers was to obtain ginger as perfectly free from the resinous matter as possible, so that the resin was got rid of first, and, as Mr. Budden had said, the delicate flavour of the ginger was really in aqueous solution, and was prepared in a very strong form for that purpose.

Mr. Budden said that he knew of an actual case where ginger, which had been thoroughly extracted with strong spirit, and which was merely a mass of cellulose, fibre, and starchy matter, with no resin left, and practically tasteless, was used for adulterating purposes, to his certain knowledge.

NOTE ON CENTRIFUGAL MILK-TESTERS.

A CONTRIBUTION TO THE DISCUSSION ON MR. EMBREY'S PAPER. (See THE ANALYST, May, 1893.)

By DR. P. VIETH.

OF all the methods which have been suggested and introduced for determining fat in milk outside the chemical laboratory, and by persons who are not trained chemists, those are now reigning supreme which are based upon the principle of decomposing the milk proteids by the action of an acid, submitting the mixture to centrifugal force, and measuring the volume of the separated fat. It is well known that the first apparatus—and for a number of years the only one—in which this principle was put into practical shape was the Lactocrite. The correctness of the results obtained by its use was fully acknowledged, but the price of the machine and the skill necessary for working it prevented its more general application. Since the last three years a number of other machines based on the same principle have been introduced, and the attention which they—or, at least, some of them—have received is shown by Mr. Embrey's paper, read before the Society of Public Analysts at the April meeting, and the discussion by which it was followed.

I must admit that, when I first read and heard about the Babcock process, I had grave doubts whether, by treating milk with an equal volume of concentrated sulphuric acid, the fat would not be attacked as well as the proteids and sugar. Experience has since shown me that this is not the case—at any rate, not to such an extent as to interfere appreciably with the results.

The first original Babcock apparatus which came over to Germany appears to have been rather a shaky machine; in a competition of milk-testers it was condemned. A second machine was imported last year, and exhibited at a dairy show at Rostock. It did not give the impression of being of sufficiently strong construction, but served as the model for constructing stronger machines. When one of the new machines was tested at the Agricultural Experimental Station of Rostock, Professor Heinrich, the principal of that station, in a long series of comparative experiments, found that the results obtained by the Babcock method were on average .22 lower than the results arrived at by gravimetric method of analysis. The Babcock made 800 revolutions per minute. Professor Heinrich then combined the Babcock tester with the driving-gear of the Lactocrite, and thereby was able to give the machine a speed of 1,500 revolutions per minute; the results now agreed with the gravimetric analysis within .03 on average. In order to reach this higher speed the machine had again to be reconstructed, by fitting to it a much larger driving-wheel, and giving it a stronger and heavier stand.

After having used this improved machine for some time, I can testify that it is now a very useful apparatus, worked with the greatest ease and without any difficulty, and giving very satisfactory results. Comparing the Babcock with the Leffmann-Beam apparatus, I found a very satisfactory agreement. I give the results of one series of experiments as an example:

Babcock	3.0	3.2	3.35	3.35	3.2	3.7	3.0
Leffmann-Beam	3.1	3.2	3.4	3.4	3.2	3.7	3.0

I have used the Leffmann-Beam apparatus regularly for six months, and like it very much indeed. It is an elegant little machine, which in my hands has always worked without the slightest hitch. Although two samples only can be treated at the time, I have found it possible to complete in a given time as many examinations as with a Babcock apparatus fitted with ten test-bottles.

DISCUSSION.

Mr. Cassal said it appeared to him that Dr. Vieth held the same view which he (Mr. Cassal) had taken when Mr. Embrey read his paper on the Lister-Babcock process—namely, that however ingenious the Lister-Babcock process might be, it ought not to be recommended for the use of persons who had had no scientific training whatever, and who were not capable therefore of drawing correct conclusions from the results observed. Unfortunately, it happened that in the number of *THE ANALYST* in which the paper and discussion appeared the report of the remarks which he (Mr. Cassal) had made was such as to make it appear that he entertained a high opinion of this machine and of the process. There was nothing in the report of those remarks, as printed, to show the opinion—which he certainly held very strongly—that this apparatus could only be used properly by trained persons. It happened that his remarks, as printed in *THE ANALYST*, merely referred to some experiments that he had made with a view of testing centrifugal machines for separating mixtures of liquids. In view of this fact, he now took the opportunity of again expressing his

opinion that the machine, and the process, ought only to be placed in the hands of trained persons. On the occasion referred to he had forgotten to mention an observation which he had made in working the Leffmann-Beam process—a process which he fully agreed in thinking was a very good one in skilled hands—namely, that while in the great majority of cases the process worked quite satisfactorily, occasionally a result was obtained which was entirely wrong. That might be due to an error in the making of the mixture, or it might be due to insufficient rotation, or to other circumstances which he had not investigated, but the fact remained that he had noticed that inaccurate results were sometimes obtained. This was in itself enough to show that processes of the kind should not be recommended for the use of people who could not really judge analytical results. He took the opportunity of recording a protest against the position that was taken up by the reader of the paper upon that occasion, and by some of the speakers; for the absence of such a protest from the report of the proceedings amounted to committing the Society of Public Analysts, and public analysts generally, to a recommendation of the process for use by untrained and unskilled persons. He also felt bound to express his deep regret that, in consequence of what took place on the occasion referred to, it might be supposed by persons who did not know the Society that it had been made use of to advertise the Lister-Babcock machine and process for the purposes named, a sort of proceeding to which, of course, the Society of Public Analysts should never, and would never, consciously lend itself.

Mr. Richmond said that Mr. Cassal informed the Society that although the results of experiments made by centrifugal machines were, as a rule, accurate, it sometimes occurred that one case was not accurate. He would like a little more information as to the latter point. Was it a considerable amount above or below the fat estimated gravimetrically, or was it that the fat could not be obtained in proper condition for reading?

Mr. Cassal replied that in some cases the amount of fat was entirely out. He was speaking from memory, but he thought sometimes as much as 0.5 per cent. out as compared with a calculated result or with the accurate determination of the fat by the Werner-Schmid or by the Adams method. So far as he could recollect, he had obtained, perhaps, some half a dozen cases of the sort while using the Leffmann-Beam process. He did not say that this was the fault of the process; it merely served to show that errors would occur, and that it would not of necessity yield accurate results automatically and invariably.

Mr. Richmond believed that there must be something in Mr. Cassal's mode of working which was not in accord with his (Mr. Richmond's) mode of working. He had checked his results by the Adams and Werner-Schmid processes, and had also checked them by the calculation method, yet he did not find that the results differed. On one occasion he had found—when using a new bottle of acid which was stronger than usual—that the fat did not separate so well, there being a considerable number of dark particles in it; but on slightly diluting the acid this difficulty disappeared. The results obtained had been so good of late that he placed absolute confidence in the method, and he would almost as soon trust to it as to the Adams method.

On the Recognition of Mixtures of Butter and Margarine, or of Butter with other Fats, whether Vegetable or Animal. A. M. Houzeau. (*L'Industrie Laitière*, 1893, 19, 147.)—If equal weights of butter and margarine are dissolved in equal volumes of alcohol of constant strength, at a known temperature, the point at which turbidity is apparent in these solutions varies with the temperature, in proportion as these fats are more or less soluble; the temperature observed at the moment when the turbidity of the alcoholic liquor is sufficiently pronounced serves to show the relative solubility of these fats comparatively.

In order to determine the point of turbidity with exactitude, the solutions are cooled in a flat-bottomed flask, on a point in which a spot the size of a wafer is made with Sabatier's ink. The same weights of butter or margarine are always used. The temperature is taken at the moment when the white spot becomes invisible. Two experiments on the same butter can be read within 0.1° or 0.2° . A difficulty arises since the degree varies in pure butters; and butters prepared at an interval of a few weeks, or even from 10 to 15 days, from the milk of the same cow show differences. This difficulty is due to the larger or smaller proportion of soluble glycerides which are present. Hence purification is necessary to secure the same degree in all butters. This purification is easily effected by dissolving in a constant amount of alcohol, warming to a temperature at which the fat dissolves, and then cooling in a bath at a constant lower temperature. The deposited matter is separated by filtration, the funnel and receiver being also in the bath. The less soluble glycerides are deposited.

When the alcohol is driven off this residue should be treated as before. The results after this purification are of a much more constant nature.

Pure butter fat before purification	63.0°
Same butter fat with 16 per cent. margarine...	68.0°
" " 12 " "	66.5°
" " 8 " "	65.5°
" " 8 " oil	65.4°
			Before Purification.	After Purification.	
Pure butter fat	59.0°
Another butter fat	61.0°
" "	64.2°
" "	67.5°
					60.0°
					60.0°
					60.2°

A second purification in the same manner as the former, but at a different temperature, produces quite constant results:

		Original.	First Purification.	Second Purification.
First pure butter	...	59.0°	...	42.0°
Second "	...	64.2°	...	42.0°
Third "	...	67.0°	...	42.0°

The delicacy of the method is seen from the following figures:

		First Purification.
Pure butter	...	60.0°
" with 6 per cent. margarine	...	60.9°
Second pure butter	...	60.0°
" with 8 per cent. margarine	...	62.6°
Third pure butter	...	60.2°
" with 8 per cent. cocoanut-oil	...	59.0°

					Second Purification.
Pure butter	42.0°
"	with 6 per cent. margarine	43.0°
"	with 8 per cent. cocoanut-oil	41.0°

The amount of volatile fatty acids purified in this manner is constant.

A mixture of margarine and cocoanut-oil could be made to evade the turbidity test, but would immediately be recognised by a determination of the volatile fatty acids.

Example.	Degree of Turbidity.	Volatile Acids.
Pure butter	42.0°	18.3 c.c.
" with a mixture of margarine and cocoanut-oil 10 per cent.	42.1°	16.7 c.c.

In this paper the author does not give details, but promises to fully describe his method in a further communication as to quantities taken, etc., at an early date.

L. K. B.

The Application of Sodium Peroxide to Analysis. W. Hempel. (*Zeit. Anorg. Chem.*, iii. 193, 194.)—Commercial sodium peroxide is free from sulphur and practically free from chlorine; it contains 60 to 70 per cent. of Na_2O_2 , the rest being sodium oxide. In the rapidity with which it effects oxidation at a red heat it far surpasses all other oxidizing agents, and loss during the operation is reduced to a minimum. The recognition of chromium and manganese by heating the suspected substance with about twice its weight of the peroxide is a matter of seconds, and the opening up of tungsten minerals with this reagent is exceedingly easy; thus, when 1 part of wolfram is melted with 4 parts of sodium peroxide, all the tungsten is converted into sodium tungstate in a few minutes. Equally expeditious is the decomposition of titanite iron by melting one part of the finely powdered ore with 4 parts of sodium peroxide; the melt is dissolved in hot water, the ferric hydroxide filtered off, the solution mixed with strong sulphuric acid, evaporated until part of the acid has been expelled, and when cold poured into a large quantity of cold water. Filtration, if not clear, and precipitation of the titanite acid from the boiling solution with sulphurous acid, conclude the operations.

For attacking sulphides the peroxide must be diluted, as the action is otherwise too violent. One part of the substance, 2 parts of soda and 4 parts of sodium peroxide are heated together in a silver crucible for a few minutes. The mass is dissolved in water, ferric oxide is filtered off, and the sulphuric acid in the filtrate precipitated with barium chloride after acidification. Lead and zinc go into the aqueous solution, so that this is at the same time an easy way of separating the iron in galenas and blendes. The following results are cited:

		By Aqua Regia.	By Fusion with Sodium Peroxide.
Sulphur in blende, per cent.	...	26.46	26.53
" pyrites "	...	36.05	36.07

A. G. B.

The Solubility of Rosin Oil and Mineral Oils and Mixtures thereof in Acetone. F. Wiederhold. (*J. Prakt. Chem.*, 1893 [2], xlvii. 394-397.)—Rosin-oil dissolves in acetone in nearly all proportions, but mineral oils are either sparingly or not at all soluble therein. Six different samples of rosin-oil were investigated in this respect, and it was found that all of them dissolved to a clear solution at 15° C. in half their volume of acetone. It is of importance that the acetone be dry and free from acid; the presence of aldehyde may be allowed. In order to keep the solvent dry, it must be exposed to the air as little as possible to avoid the condensation of moisture, which always occurs in vessels containing such volatile solvents, by the lowering of the temperature below the dew-point, owing to the latent heat of vaporization of the liquid. The difference between the solubility of rosin-oil in moist acetone and in dry acetone is very considerable, so much so, indeed, that this forms a rapid test for moisture in the solvent.

Russian mineral oils require very varying quantities of acetone for their solution, and the amounts are apparently relative to the specific gravities of the oils. Thus, for perfect solution at 15° :

One volume of	Sp. Gr.	Requires of Acetone.
Burning oil ...	0.830	4 vols.
Russian spindle oil ...	0.898	40—41 „
Oleonaphtha ...	0.908	70—71 „

But, as might be expected, considering that the commercial mineral oils are of varying composition, the quantity of acetone necessary varies between considerable limits.

As a means of identification, the solubility may be applied by shaking 2 c.c. of the sample with 20 c.c. of acetone in a mixing cylinder, graduated to $\frac{1}{10}$ c.c., and reading the portion left undissolved after 12-24 hours. In this way the following figures were obtained :

	Sp. Gr.	Left undissolved at 15° C.
1. Russian cylinder oil (Nobel) ...	0.913	1.7 c.c.
2. Russian "Distillate" (Nobel) ...	0.910	1.5
3. Mineral oil, No. 1. (Schibaeff) ...	0.908	1.5
4. Caucasine 1 (Nobel) ...	0.808	1.5
5. Oleonaphtha A.A. (W. Schliemann)...	0.905	1.5
6. Bakunit (Deneys and Co.) ...	0.905	1.5
7. Russian spindle oil (Nobel) ...	0.898	1.3
8. Pale machinery oil (Thompson and Bedford)	0.905	1.1
9. Extra spindle oil (Thompson and Bedford)	0.885	0.8

The American cylinder oils are almost entirely insoluble in acetone.

Viscid and dark, unrefined oils must be treated at a higher temperature, for which purpose the mixing cylinder may be heated in boiling water; the figures thus obtained cannot of course be compared with those obtained at 15° C.

Care must be taken that the oil tested is not acid. Referring to the detection of acidity in an oil, the author points out that the test originally devised by him remains the best. The oil is shaken with cuprous oxide (not cupric oxide, as stated in the text-books), which will form a copper soap, with separation of metallic

copper, if free acid be present; this soap will dissolve in the oil and colour it green, but judgment must not be passed until after the lapse of twelve hours.

The solubility in acetone will serve for the detection of rosin-oil in a mineral oil. As is well known, this sophistication may be effected by mixing the rosin-oil with a mineral oil of lower specific gravity than that stipulated in the specification, for the specific gravity is raised by the rosin-oil. As an example, the author quotes the case of an "oleonaphtha" which fulfilled the specified specific gravity of 0.906-0.908, but consisted of a Russian spindle oil, to which had been added one-sixth of its weight of red rosin-oil; this sample left only 1 c.c. undissolved when 2 c.c. were shaken with 20 c.c. of acetone, as above described, whereas a normal oleonaphtha should leave 15 c.c. undissolved.

Finkener suggested that a mixture of 10 volumes of alcohol (sp. gr. 0.818) and 1 volume of chloroform should be used for detecting rosin-oil in mineral oils, in the proportion of 10 volumes of the mixture to 1 volume of oil at 23° C., its application being similar to that suggested above. The necessity for a temperature of 23° renders this proposal impracticable as it stands; but the author finds that it will answer at 15° if 16 volumes of the alcohol-chloroform mixture be substituted for 10 volumes.

A. G. B.

[The author appears to be ignorant of the fact that Demski and Morawski have suggested the use of acetone for detecting rosin-oil in mineral oils. Compare Allen's "Commercial Organic Analysis," ii. 466.—A. G. B.]

REVIEW.

GLI OLII (OILS). By G. de Negri and G. Fabris. Publication of the Laboratorio Centrale delle Gabelle, Rome.

Part I. Sulle reazioni che caratterizzano l'olio di oliva. (On the reactions that characterize olive-oil.) 1891.

Part II. Sulle reazioni che caratterizzano alcuni olii di semi. (On the reactions that characterize some seed-oils.) 1893.

The work, which emanates from the Laboratorio delle Gabelle—the Somerset House of Italy—contains nearly all that is known about the oils treated of. The authors have determined the density, freezing-point, melting and solidifying points of the fatty acids, rise of temperature with sulphuric acid, iodine absorption and potash absorption, and they have also examined the various colour and other reactions for specific oils. The results obtained are classified under the various oils, and a *résumé* is given of the work of other observers. Typographical errors occur in a few instances, the most objectionable being the substitution of 6 for 9 in several places.

The authors adopt Jean's thermo-oleometer for the determination of the rise of temperature with sulphuric acid. This is a great advance, as there can be no doubt as to the meaning of this determination; it would be a great boon if this method or that of Thompson and Ballantyne were officially adopted. We are glad to see that the authors have definitely disproved that the presence of sulphur is characteristic of

oils derived from the *Cruciferae*; they also show that the oils of bitter almonds, apricot, peach, and cherry do not contain prussic acid if obtained by cold pressure, while if these oils are extracted by solvents this acid occurs. They have studied Rénard's method for the detection of earth-nut-oil; they modify it somewhat as follows: The washing with ether of the lead salts is done as far as possible in the beaker in which they were precipitated, and finally they are collected on a filter; the filter is placed in a separating funnel, at the bottom of which is a small wad of glass-wool to stop the filter-paper from running through; dilute hydrochloric acid and ether are added; after the lead-salt is decomposed, and the fatty acids dissolved in the ether, the aqueous portion is first run off, and then the ethereal solution collected in a beaker, the ether evaporated, and the fatty acids dissolved in alcohol as usual. The quantity of arachidic acid found in earth-nut-oil was from 4.4 to 4.8 per cent., and the authors were able to estimate 10 per cent. of earth-nut-oil in olive-oil, working at 15° C.; with smaller quantities the results were not reliable.

A study of the little-known oil of coffee was made. Maize-oil was also examined.

We can recommend this work not only as a book to be read, but also as a good work of reference. We congratulate the authors on having produced a work of such value.

H. D. R.

IMPORTANT APPEAL CASE.

IS COLOURED ACETIC ACID VINEGAR?

(From the *Birmingham Post*.)

AT the Birmingham Quarter Sessions on Monday, July 3, before the Recorder, Mr. J. S. Dugdale, Q.C., William Kirby, 69, Digbeth, appealed against a conviction made by Messrs. W. N. Fisher and H. P. Ryland, Justices of the Peace for the city, on May 19, for an alleged offence under the Food and Drugs Act. Mr. T. Terrell (London) and Mr. Foster (instructed by Jaques and Sons) appeared for the appellant, and Mr. Hugo Young and Mr. Symonds (instructed by Mr. Bell from the Town Clerk's office) for the respondents.

Mr. Hugo Young, in opening the case for the respondents, said the appeal was against a conviction by the Birmingham magistrates, under Section 6 of the Food and Drugs Act, 1875. He said that on April 13 Inspector Davis, in pursuance of his ordinary duties, went to appellant's shop and demanded half a pint of vinegar, for which he paid one penny, the price of best vinegar. After the purchase Davis observed the particulars required by the Act before the vinegar was submitted for analysis. The Public Analyst reported that the liquid contained 70 per cent. of pyroligneous acid, and consequently proceedings were taken. The question arose in that case What was properly sold under the designation of vinegar? In order to go back to the commencement he thought it was obvious to everybody that the term vinegar was derived from *vin aigre*, the French for sour wine. He believed that wine allowed to go sour under atmospheric conditions would produce vinegar, and no doubt that was the origin of vinegar known at the earliest time. In producing vinegar they must have first of all alcohol which was converted by atmospheric oxygen by the process called acetous fermentation into acetic acid. In the course of the production of that acetic acid, the various properties of the wine remained, along with the acetic acid which was produced by the conversion of the alcohol, and therefore what they had to see was what vinegar consisted of. He thought it was right, from their point of view, it should consist of dilute acetic acid, plus the other properties of the wine. They defined vinegar as a liquid consisting essentially of impure dilute acetic acid obtained by the oxygenation of an alcoholic liquid. The real point was whether, without that alcohol as the basis—without which they did not get the other properties of vinegar—it was right to call the liquid vinegar. It had been said that these prosecutions, if upheld, would interfere with a large industry, but there was no desire to interfere with the sale of acetic acid

made from the distillation of wood. If this liquid served the purposes of vinegar then the public would buy it, and there would be no reason for selling it as vinegar. At present it was sold under a designation it did not properly bear, and the only object could be to mislead the public. If in the appellant's case the liquid sold was better than vinegar, why not sell it under its distinctive term, and let the people know what they were buying?

Thomas Davis, Inspector under the Food and Drugs Act, deposed to purchasing the vinegar from Mrs. Kirby. He informed Mrs. Kirby that the liquid was for analysis, and she then said that it was not malt liquor. She also replied that she did not mind, as she had had a letter from the firm that supplied it, saying they would be responsible for any proceedings which might be taken against her husband for selling it.—Cross-examined: He went to appellant's shop on his own account. He had previously noticed that there had been prosecutions in different parts of the country, and was aware the question had been raised as between vinegar and malt vinegar. He did not ask for malt vinegar purposely. He was unaware that there were in the trade distinctions between vinegar and malt vinegar.

Dr. Alfred Hill, Medical Officer of Health and Public Analyst, gave a description of his analysis and the composition of the liquid submitted to him. True vinegar, he said, was made from the juices of plants, fruits, sap, or any material extracted by water. Pure acetic acid could be produced in a cheaper form than by getting it from alcohol. The common source of obtaining acetic acid for commercial purposes was from wood.—Cross-examined: Malt converted into alcohol, then acetified, and then distilled, was in his opinion distilled vinegar. It was not vinegar in the ordinary sense.—Mr. Terrell: Do you know that persons who produce vinegar in that way are called vinegar makers by Act of Parliament?—Witness: Distilled vinegar makers.—Mr. Terrell: No, vinegar makers.—Witness: I should apply the qualifying adjective.—In answer to further questions, witness said that he did not know of any vinegar maker who sold vinegar uncoloured. He believed so-called malt vinegars were coloured with caramel.—Mr. Terrell: Do you think that adulteration?—Witness: I don't call it adulteration; I think it is done simply to pander to a vitiated public taste.—You are going to put that vitiated taste right? I leave that to you.—He did not think that pure vinegar made from malt was unsuitable for the manufacture of pickles, and he did not know that such vinegar was dosed with sulphuric acid to prevent it going musty. The so-called vinegar made from wood was not so liable to go musty. Witness had certified that the vinegar sold to Davis contained 70 per cent. of pyroligneous acid, but it really contained more. He had given the vendor the benefit of every weak point.—Mr. Terrell: Was not the real reason you certified the 70 per cent. that you did not analyze the vinegar at all?—Witness: That's an insult, and I decline to answer.—Mr. Terrell: Was not the real reason that you only distilled off the acetic acid and weighed the residue?—Witness: I decline to answer the question. I don't come here to be insulted.—Mr. Terrell: I am not insulting you.—Witness: Then your code of morals is different to mine.—The Recorder pointed out to the witness that counsel was asking him how he conducted the analysis, and witness replied that he would answer if counsel were courteous.—Mr. Terrell: Did you do anything more than distil off the acetic acid?—Witness: You know I did. I will repeat it for you, since your memory is so short. Witness detailed the method of his analysis.

Mr. Terrell admitted that the liquid in dispute was made from wood, and the question was whether it came under the term "vinegar" or not.

Mr. Otto Hehner, Mr. Jones (Wolverhampton), and Mr. A. H. Allen (Sheffield and London) gave evidence, and agreed that the liquid in question could not properly be called vinegar.—In cross examination. Mr. Allen said that as a food there was little difference between the malt vinegar and the liquid in question, but as a stimulant to latent digestion there was a difference. Several grocers and drysalters also gave evidence, and said that when they were asked for vinegar they supplied malt vinegar.—William Singleton, formerly a maker of pyroligneous acid, said that he believed the difference in cost of making acetic acid as distinguished from malt vinegar was between 1s. 6d. and 6d. per gallon.

For the appellant, Mr. Terrell said that the case was one of great importance, because that was the only tribunal before which that matter could be brought as a matter of final appeal. He took it that it must be a question of fact whether that was vinegar or not, and therefore one upon which it would be difficult, at any rate, to state a case. The trade which was attacked had grown for at least seventy or eighty years, and during that time a great industry had been developed in manufacturing acetic acid from wood for the purpose of vinegar making. Extensive works had been constructed in South Wales and elsewhere, and an enormous amount of capital had been invested in these works. Up to about three months ago the trade had remained absolutely unchallenged. The question to be determined was really what was the scope of the word "vinegar." He was going to suggest, and asked the Court to find, that any

potable form of acetic acid might be defined as vinegar. That there were several different forms of acetic acid there could be no question. Step by step science had progressed, and new discoveries had been made, among them being that of the making of vinegar from the destructive distillation of wood. This industry had grown up under the eyes of the law, and had been openly conducted for many years. He should call evidence to show that the liquid in question was absolutely better than vinegar made only from malt or malted grain.

Professor Attfeld, F.R.S., of Watford, and 17, Bloomsbury Square, London, editor of the *British Pharmacopœia*, said that in his opinion there was no one fluid that could claim the word "vinegar" for a name. Vinegar belonged to a class, and was a generic term. He was familiar with vinegar made from wood, and when that substance was properly prepared, it could legitimately be called vinegar. He was acquainted with the manufacture of vinegar, and was consulted by various vinegar manufacturers. To his knowledge there was no so-called malt vinegar properly made now. He looked upon the term "malt vinegar" at the present time as a misnomer, and he should call malt vinegar vinegar made from malted grain alone. The malt vinegar commonly so called was not, strictly speaking, malt vinegar. With regard to the substance in dispute, he had known of it for nearly forty years, and considered it came under the generic term of vinegar. It was not fair to call it pyroligneous acid. He considered the dietetic value between malt vinegar and the liquid in dispute was absolutely insignificant. There was nothing injurious to health in the substance, as far as he was aware; it fulfilled all the uses of vinegar.—Cross-examined: He could not give an exhaustive, inclusive and exclusive definition of vinegar. His definition was acetic acid and water sufficient to make it potable.

Dr. Suckling said that he had heard the evidence of Professor Attfeld and generally agreed with it.

Mr. H. Grimshaw, Manchester, and Mr. F. Sutton, Yarmouth, also gave evidence.

Mr. J. Taylor, of Torquay, said that dilute acetic acid had been sold as vinegar for very many years. It was very pure, and was superior to malt vinegar by reason of its containing neither sediment nor vegetable matter, the latter being liable to putrefaction. By Mr. Young: He preferred the acetic acid coloured to the malt trade vinegar, and thought that the term vinegar was justly applied to it. By the Recorder: He had heard of champagne being made in England. If it were not made from the grape he should not call it champagne. Mr. Henry Evans, Inland Revenue officer, stated that the Somerset House authorities recognised pyroligneous acid as vinegar, and compelled all manufacturers of it to hold licenses.

Mr. B. Biggs, of London, drysalter, stated that since the beginning of this century vinegar had been made from acetate of soda. To his mind any acid with a pleasant flavour would be vinegar. The Recorder: Is it the flavour, then, that makes the vinegar? Witness: It depends upon the person's taste. In reply to further questions, the witness said that in preparing red herrings and hams pyroligneous acid was used.

Mr. Woolway, of the City Vinegar Company, Bristol, said that he had made vinegar from pyroligneous acid, and took five prizes with it at the Paris Exhibition. By Mr. Young: Witness in reality was the appellant in this case. Probably the public would not buy the diluted acetic acid so readily if they knew what it was.

Evidence as to the long-continued practice of selling the substance in question as vinegar was given by Mr. Robert Hemming, of Summer Lane, and Mr. S. Main, of Bromsgrove Street, grocers, the witnesses stating that it had always given satisfaction to their customers.

Mr. Terrell, summing up the appellants' case, said that his clients had proved by the highest scientific evidence that, as far as dietetic or food value went, the vinegar in question was equal in every practical respect to that made from malt or grain. Vinegar, he contended, was a generic term to be applied to such substances which were sold as vinegar, and answered the purpose of vinegar, but when one wanted to distinguish one kind of the liquid from the other one must describe what he particularly required. Those who wanted wine vinegar would ask for it; those who wanted grain vinegar would say so, and so on. Vinegar made from pyroligneous acid had been sold for many years, and the makers of the malt-grain vinegar had no exclusive right to the term vinegar. The Act of George III. showed that "vinegar" and "acetic acid" were synonymous terms, for the Act made a person a vinegar-maker the moment he began to purify and rectify acetic acid, and brought himself within the cognisance of the excise. This trade had been in existence for a hundred years, or thereabouts, and its growth had never been attacked till a few months ago. The business had been recognised by legal decisions, and had submitted to the operation of the excise duties upon vinegar. The suggestion that it should be called by some other name than the one under which it had long been regularly sold was a suggestion that would apply to many other articles of commerce. It would be a very serious thing if the Court should come to the conclusion that this was an ille-

gitimate and fraudulent trade, and if the appellants should be compelled to sell their product under another name than that under which they had legally, as he contended, sold it so long.

Mr. Young, in his reply on the whole case, said that the object of such prosecutions as this was that when people asked for one thing they should not be given another. The contention that the defendants were recognised in certain Acts of Parliament for excise purposes as vinegar-makers, and that therefore what they sold as vinegar would, if sustained, enable any person who treated acetic acid in such a way as to come under the excise to sell any of his products, even pure acetic acid, as vinegar. The appellants' case really was that they only were entitled to call their product vinegar, and that the real malt vinegar must be given some specific name.

Mr. Terrell said that the appellants had repudiated this construction of their case. What they contended was, a person asking for vinegar might lawfully be supplied with either of the different kinds.—Mr. Young said that, without putting the words into his friend's mouth, he was quite entitled to say that the effect of their arguments being admitted would be what he had stated. He asked the Court to say that if these people wanted to carry on this trade, with which nobody desired in the slightest degree to interfere, they must carry it on honestly and straightforwardly. When the appellants admitted, as they had admitted, that to get the public to take it was necessary to give it a false name, and that the public would not take it under any other name, their case was gone. He contended that the term vinegar must be limited to the fermented article, which contained something besides acetic acid, and that acetic acid was not entitled to go through the world with two names, at one time being described as acetic acid, and at another being allowed to masquerade as vinegar.

The Recorder said that he saw no useful purpose to be served by reserving judgment. Having listened very carefully for a day and a half to the arguments so ably adduced on both sides, he thought he was in a position to give his decision at once. The question really was whether the substance which had been sold on this occasion by the appellants could be allowed to bear the name of vinegar. If it ought not to be sold as vinegar, then there had been sufficient prejudice to the purchaser to justify a conviction under the Food and Drugs Act. There was no dispute as to the constitution of the substance. As to whether it was entitled to be called vinegar, counsel for the appellants had, with great skill and perseverance, endeavoured to establish the affirmative on three grounds. The first ground was, that the substance was recognised by law as vinegar. Without going through the Acts of Parliament and the decisions that had been quoted, he (the Recorder) would say that he had come to the conclusion that the contention that vinegar and preparations of acetic acid were in law the same thing could not be accepted. The object of the Acts of Parliament quoted was to take care that certain excise duties were enforced, and for that purpose they enacted that persons who had or received into their custody and possession, amongst other things, any vinegar or acetic acid, or other matters for certain purposes, should be deemed vinegar-makers. Those enactments did not mean to enact that acetic acid should be vinegar, or that the makers or purifiers of acetic acid or pyroligneous acid, although they were to be deemed vinegar-makers for the purpose of these Acts, should be entitled to have the acetic acid which they produced termed vinegar. He thought that the words "deemed to be vinegar-makers" showed conclusively that it was for the purpose of these duties that they were to be taken under the generic name "vinegar-makers," which included a number of things, and he could not hold that it had been recognised by these various Acts of Parliament that dilute acetic acid, coloured, was vinegar. The decisions that had been quoted, and which had reference to excise questions, he interpreted in the same manner, and therefore the argument depending upon recognition by law was unfounded. As to the second point, whether this mixture was entitled by long use to be termed vinegar, evidence had been given that for a great number of years it had been sold as vinegar. On the other hand, it was admitted that the public had not known that it was merely dilute acetic acid coloured that they were buying, and that it had not been discovered by the public till quite recently, when the analysts had found out what the exact constituents of the substance were. Therefore he did not think that this substance had made out its title by long usage to be recognised as vinegar. The third point made by the appellants was that the substance was really vinegar in point of fact. It was clear that before the invention of this substance the original definition of vinegar was a vegetable juice or infusion which had passed through the alcoholic and acetic fermentations. In the "British Pharmacopœia" Professor Attfield defined vinegar as a substance "prepared from the mixture of malted and unmalted grain by acetic fermentation." He was surprised, therefore, to hear Professor Attfield say on Monday that this definition of vinegar, for which he was responsible in so important a publication as the "British Pharmacopœia," was put in simply for the preparation of scap-plaster. On the very same page of the book the Professor,

alluding to vinegar, stated what the dose was to be; and he could not believe that a gentleman of eminence who was responsible for so important a publication could have been guilty of what would have been unpardonable carelessness of putting in such a definition of such an article as vinegar simply for the purpose of soap-plaster. But in the "Pharmacopœia," in addition to defining vinegar, Professor Attfield had also defined dilute acetic acid. Therefore he (the Recorder) came to the conclusion that as far as Professor Attfield was concerned, the definitions of vinegar and of dilute acetic acid were distinct. Reviewing the other expert evidence, the Recorder pointed out that Mr. Allen had said that dilute acetic acid not produced from any process of fermentation was not true vinegar; but was "as different from it as silent spirit is different from wine." In fact, in the evidence one might just as well be called upon to decide that alcohol and water was wine as that coloured acetic acid and water was vinegar, for alcohol was as much the essential of wine as acetic acid was the essential of vinegar. Therefore the case of the appellant seemed to be a *reductio ad absurdum*. Under these circumstances, he was forced to the conclusion that what was sold in this case was not entitled to the name of vinegar. He did not think there was very much weight in the arguments of the great disturbance of trade that would be caused by a decision adverse to the appellants. In a case of that kind the law could not take that into consideration at all; but he would point out that a similar argument was used with regard to margarine. It was said that the public would cease to buy margarine if that substance were given its proper name, but it was common knowledge that the margarine trade was now in a more flourishing state than ever before. He had no doubt that his decision in this case—which, if it should be confirmed elsewhere, would have a far-reaching effect—would cause a good deal of disturbance and inconvenience to the manufacturers of this article. The substance, however, was no doubt extremely valuable for many things, and served the purpose of vinegar in many ways, and he could not help thinking that the discussion which had been raised by this case must have shown the public the value of the substance for what it was as well as what it was not. The article appeared to be a valuable thing in itself, and would find its place in commerce, and therefore he did not think that in the end the manufacturers of it would be at any loss. But it was not entitled to be called vinegar, and consequently the appeal must be dismissed, with costs against the appellant.

CORRESPONDENCE.

To the Editors of THE ANALYST.

SIRS,—In THE ANALYST for July last, p. 171, Messrs. Richmond and Boseley state that for the estimation of mixtures of milk-sugar and cane-sugar in milk, "Stokes and Bodmer's method (ANALYST, X., 62) is suitable, but probably not very reliable." (Cf. Hehner, ANALYST, VI., 218.)

On turning to the November number of THE ANALYST for 1881 we find Mr. Hehner's paper on sugar titration, which also was fresh in our minds when we devised the process referred to. Mr. Hehner's experiments are very interesting, as showing that if you replace the necessary caustic potash and Rochelle salt of our solution by sodium acetate, sodium carbonate, or ammonium chloride, you get erroneous results. But they in no way show that the solution containing, as advised by us in THE ANALYST, X., 62, copper sulphate, caustic potash, Rochelle salt and ammonia, does not give reliable results. Mr. Hehner's paper is especially useful in pointing out that you cannot vary the ingredients without varying the results. He lays stress equally on the fact that Fehling's solution itself "only gives correct results when similar conditions are as nearly as possible adhered to." We still frequently use the method for the estimation of sugar in milk, sugar mixtures, and sugar in urine, and find it exceedingly reliable, rapid, and accurate. Other observers also testify to its reliability. We object to our method being, without trial, condemned as "probably not very reliable," merely because a different solution to that which we recommend gave inaccurate results some years before.—We remain, yours faithfully,

ALFRED W. STOKES.
R. BODMER.

ERRATA.—Page 183, foot-note, for $(C_2H_2O)_2O$ read $(C_2H_3O)_2O$; for 353 read 353; and for $(C_2H_2O)OH$ read $(C_2H_3O)OH$.

THE ANALYST.

SEPTEMBER, 1893.

ON THE RESULTS OF THE WORKING OF THE ADULTERATION ACTS IN DUBLIN.

By SIR CHARLES A. CAMERON, M.D.

(Read at the Meeting, July 18th, 1893.)

ON August 6, 1860, the first general statute for preventing the adulteration of articles of food and drink was passed by Parliament. It provided for the appointment of public analysts, but not for inspectors of food.

The first public analyst appointed under this Act was the late Dr. Henry Letheby; he was elected to that office by the Corporation of London. The next appointment was made by the Corporation of Birmingham, and the gentleman selected by them, Dr. Alfred Hill, is still in office. I was the third public analyst appointed, having been elected by the Corporation of Dublin, on October 3, 1862.

The Act of 1862 was practically inoperative in every place save Dublin. Some years ago, having made inquiries on this subject, I found that there were no prosecutions instituted under its provisions, except in this city. Shortly after I was appointed I suggested that the inspectors of the markets should purchase articles of food on their own behalf, and submit portions of them to me for analysis. For several years they purchased various kinds of food, and when they were found to be adulterated the vendors were prosecuted. The procedure was simple enough. The purchaser informed the vendor that he was about to submit the article purchased to the public analyst, and that the vendor might, if he chose, accompany the purchaser to the public analyst, in order to witness its delivery. On hearing a case under the Act, the justice might cause a portion of the article purchased to be analyzed by a skilful person, for in those days there was no appeal to Somerset House.

On looking into the records of the Corporation of Dublin, I find that in the first year after my appointment there were 46 articles of food and 11 drugs analyzed. Out of 19 specimens of milk examined there was only 1 pure; the others were adulterated with from 20 to 60 per cent. of water. Eight samples of flour were analyzed, and 1 of them was found to be adulterated with potato flour. Out of 9 specimens of bread, 4 were adulterated with potatoes, rice flour, and alum. One

of two specimens of Bermuda arrowroot consisted wholly of potato starch. The only specimen of coffee tested contained 30 per cent. of chicory. Three of the drugs were adulterated. Thus it will be seen that 50 per cent. of the articles examined were adulterated.

In 1870 the fines inflicted on food adulterators amounted to £158 10s. Amongst the persons convicted were ten confectioners. They had sold confections containing poisonous pigments, and terra alba, or white clay. Some of the lozenges sold by these confectioners contained nearly half their weight of clay. One specimen of sugar-stick contained 1 per cent. of red sulphide of mercury. All the yellow confections were coloured with chromate of lead. Since the year 1871 I have never met with adulterated confections in Dublin.

In 1872, the last year the Act of 1860 remained unamended, the number of convictions for the sale of adulterated food was 67, and the amount of fines imposed was £306 10s. During the ten years that the first food adulteration Act was in force in Dublin, there were hundreds of convictions obtained under it, notwithstanding its defects. The articles found to be adulterated were milk, butter, tea, coffee, cocoa, mustard, flour, bread, arrowroot, confections, sugar, rum and wine. The milk was nearly always adulterated, not only with water, but occasionally, though rarely, with other substances, namely, sugar, starchy matter, and salt. The butter contained excessive quantities of water and salt. On one occasion a sample was examined which contained the pulp of swedish turnip; and on another occasion a roll of butter was found to contain a snowball as its core. On several occasions tea was found to be adulterated with exhausted tea-leaves, the want of substance in it being to some extent disguised by the addition of some astringent body, such as catechu. As a rule coffee was mixed with chicory, burnt sugar, roasted grain, ground toast of bread, etc. In 1874 16 persons were convicted for selling adulterated coffee. In two instances the coffee was mixed with 90 per cent. of chicory, and in four other cases with 70 per cent. and upwards. Cocoa was chiefly composed of starch and sugar. Mustard was never found to be pure. It was always found mixed with large percentages of flour or starch, and even sulphate of lime was used to adulterate it. Flour was adulterated with rice and alum, and bread with rice, potatoes and alum. Confections were adulterated with white clay and coloured with chromate of lead, vermilion, Prussian blue, and, though rarely, with arsenical green. On only two occasions was sugar found to be adulterated, and then it was with flour. Rum was bought which, in reality, was whisky mixed with treacle. I frequently met with the spurious wine termed Hambro' sherry. What was sold as French brandy sometimes proved to be corn spirit, flavoured with various essences.

One good clause in the Acts of 1860 and 1872 was repealed by the Act of 1875, namely, that which enabled the justices to advertise a second conviction in the newspapers, or otherwise, and at the expense of the offender. Advertisements of this kind frequently appeared in the Dublin newspapers. In the front page of the *Irish Times* for February 27, 1872, the following advertisement appears, much "displayed" and in large type:

"PUBLIC ADVERTISEMENT.

ADULTERATION OF MILK.

At the Northern Police Court,
On Saturday, the 17th of February, 1872,
John Doyle, Dairyman,
Of 127, Dorset Street,
Was Fined Five Pounds
For Selling Milk
Adulterated with Fifty per Cent. of Water.

And it being his second conviction for a similar offence, the publication of this advertisement was ordered by the magistrate who heard the case. In addition to the above penalty, cost of advertisement to be defrayed by said John Doyle."

After the passing of the Acts of 1872 the adulteration of many articles of food gradually died out. Tea, flour, bread, mustard and whisky are now never found to be adulterated. Coffee is rarely adulterated, though three cases of adulterated coffee have recently been before the courts. The adulteration of milk is very much less now than formerly. Butter, however, has lately been much subject to adulteration, and I regret to say that the adulteration sometimes takes place in Ireland. With the exception of milk and butter, the food sold in Dublin is, with rare exceptions, pure, a fact which undoubtedly is attributable to the working of the Anti-Adulteration Acts.

With the view of comparing the work done in Dublin in reference to carrying out the Anti-Adulteration Acts with that accomplished in other towns, I have ascertained the number of convictions under the Provision of the Sale of Food and Drugs and the Margarine Acts in 49 towns. The results are shown in the following table:

		FOOD AND DRUGS ACT.			MARGARINE ACT.		
TOWN.	Population.	Convictions.	Fines, etc.			Convictions.	Fines, etc.
LONDON—			£	s.	d.		£ s. d.
Islington ...	319,433	36	47	5	6	Nil	Nil
Saint Pancras	234,437	31	77	9	0	Nil	Nil
Hackney ...	229,531	22	53	12	0	8	8 17 0
Southwark ...	202,537	22	20	12	6	2	9 7 0
Kensington ...	166,321	32	71	9	6	Nil	Nil
Holborn ...	141,544	3	11	0	0	Nil	Nil
Shoreditch ...	124,009	7	19	6	6	Nil	Nil
Paddington ...	117,838	7	9	8	0	Nil	Nil
Chelsea ...	96,272	15	84	16	6	Nil	Nil
Whitechapel...	74,462	Nil	Nil	Nil		Nil	Nil
Croydon ...	82,548	2	6	0	0	Nil	Nil

		FOOD AND DRUGS ACT.			MARGARINE ACT.				
TOWN.	Population.	Convic- tions.	Fines, etc.			Convic- tions.	Fines, etc.		
			£	s.	d.		£	s.	d.
Liverpool...	513,790	115	263	18	8	21	23	19	0
Manchester...	510,998	66	174	3	0	32	43	12	0
Birmingham...	483,526	32	29	17	0	15	12	0	0
Leeds...	375,540	15	42	10	0	3	6	5	0
Sheffield...	329,585	20	41	7	9	4	3	8	6
Bristol...	223,592	16	59	10	0	4	4	10	0
Bradford...	219,262	3	4	5	0	2	2	12	0
Nottingham...	215,395	5	10	0	0	1	1	10	0
Kingston-upon- Hull...	204,750	10	16	12	6	Nil	Nil		
Salford...	201,508	24	25	11	0	9	7	17	6
Newcastle - upon - Tyne...	192,205	7	3	15	0	Nil	Nil		
Leicester...	180,066	2	1	0	0	Nil	Nil		
Sunderland...	158,642	Nil	Nil			Nil	Nil		
South Shields...	141,493	1	3	0	0	Nil	Nil		
Oldham...	134,221	3	3	0	0	Nil	Nil		
Blackburn...	122,238	2	Nil			Nil	Nil		
Brighton...	116,426	5	11	10	0	1	10	0	0
Birkenhead...	101,264	4	9	10	0	5	9	0	0
Norwich...	100,970	Nil	Nil			Nil	Nil		
Huddersfield...	96,599	4	0	13	6	4	1	0	0
Derby...	95,908	11	20	9	0	3	14	4	0
Swansea...	92,344	12	19	5	0	Nil	Nil		
Gateshead...	88,588	4	5	0	0	Nil	Nil		
Plymouth...	85,610	14	60	6	0	Nil	Nil		
Halifax...	84,097	2	1	13	6	Nil	Nil		
Wolverhampton...	83,519	2	2	10	0	Nil	Nil		
Middlesborough...	76,293	4	18	18	0	Nil	Nil		
York...	72,885	2	3	9	6	Nil	Nil		
St. Helens...	68,628	6	5	0	0	11	5	17	6
London City...	68,345	1	5	6	6	Nil	Nil		
SCOTLAND—									
Glasgow...	669,059	18	38	3	8	26	37	11	0
Edinburgh...	264,787	8	13	15	0	Nil	Nil		
Dundee...	155,025	Nil	Nil			Nil	Nil		
IRELAND—									
Belfast...	265,123	22	26	3	6	9	17	2	6
Cork...	75,345	1	0	10	0	Nil	Nil		
Limerick...	37,155	Nil	Nil			7	24	0	0
Londonderry..	33,200	8	7	0	0	Nil	Nil		
TOTALS...	8,676,913	626	1,328	13	1	167	243	13	0
DUBLIN...	245,001	96	201	9	6	44	199	10	0

From January 12, 1893, up to July 13 there have been 62 convictions under the Sale of Food and Drugs Act, and the fines were £187. Under the Margarine Act the

convictions were 38 and the fines £179 10.—Total convictions, 100; total fines, £316 10s.

DISCUSSION.

The Chairman (Mr. Hehner) said that there was no one more qualified to deal with this subject than Sir Charles Cameron, who had a very large district under his control. It was a fact to be regretted that Irish butter had more water in it than the butter furnished by other European countries and New Zealand. Irish farmers continually made excuses for this—excuses which might have a certain small amount of foundation, but which he thought mostly had no foundation at all—excuses such as the state of the weather, and the feeding of the cows, and the breed of the cows. But his strong impression was that the real cause was the ignorance of the farmers in the art of making good butter. He took it that butter was one of the most important of the Irish industries, probably the most important of the future if not of the present time. Irish butter had not for many years stood in such high repute in the English market as other butters scientifically made, such as that of Denmark, the most admirable of all. He (the speaker) would urge most strongly on Irish farmers that the butter industry should be brought to a pitch of perfection similar to that which the Danes had managed to reach in the course of a few years. There an intelligently-advised Government had taken the matter in hand, and in a country where the butter industry had been exceedingly small, and which climatically was no better off than Ireland, they had, assisted by Government aid, and by the industry of the people, within a few years created a butter of the most excellent quality. He took it that if the Irish were willing to be helped, and the Government were willing to help them, that could be accomplished in Ireland also; and if that could be accomplished it would be of more consequence to the country than any other measure that could be conceived. As to the Margarine Act, he agreed with Sir Charles Cameron that in England the Act had been badly enforced, and that Dublin was a place where the Act had been enforced with something like rigour. While, of course, this was in large measure due to the energy of Sir Charles Cameron, he must be congratulated on having a Corporation behind him eager to give him advice in the matter.

Mr. Avon desired to know from what source Sir Charles Cameron derived his statistics.

Professor Tichborne differed from the chairman (Mr. Hehner) in ascribing the presence of large quantities of water in Irish butter to ignorance on the part of the farmers. He thought that the farmers quite knew what they were doing; but it was a very false economy. But lately there had been a great improvement, largely due to the starting of co-operative societies in this Country. Of course, under the old system it was impossible for the small farmer, with his small quantities, to equal the quality turned out by the Dutch producers with their high-class machinery and freezing apparatus. But now they had started in the direction of co-operative societies, and, though only two or three years old, they were spreading to an immense extent in Ireland. The result was that the butter had been largely improved in quality;

and the butter merchants themselves were associating together into societies simply for the purpose of estimating the amount of water in the samples which they bought, so that they should not pass on any quality of butter that would contain more than 15 or 16 per cent. of moisture. He had no doubt that in a few years Ireland would be, as it was now beginning to do, competing with the very finest quality of Danish brands.

Sir Charles Cameron said, in answer to Mr. Avon, that the statistics he had compiled were obtained from the town clerks of the various towns included in the table.

SIMPLE APPLIANCES FOR TESTING THE CONSISTENCY OF SEMI-SOLIDS, WITH NOTE ON A NEW METHOD OF EXAMINING BUTTER.

BY CHARLES E. SOHN, F.I.C.

(Read at the Meeting, July 18th, 1893.)

THE use of the viscosimeter, which measures the rate of flow of a liquid through an orifice, is limited to substances which are either liquid at ordinary temperatures or assume the liquid condition (without undergoing chemical change) when a moderate degree of heat is applied to them. A large number of technical products exist which do not lend themselves to this method of gauging their physical properties; for instance, semi-fluid compositions holding solid matter in suspension, paints, varnishes, and tarry compounds, emulsion-like mixtures of high viscosity, gelatinous substances and semi-solids generally. In testing the consistency of such as these, the following forms of apparatus may be of service.

Legler, some years ago (see *Chemiker Zeitung* 8, 1857; and Benedikt, *Analyse der Fette und Wachsarten*), described an appliance for measuring the consistency of the elaidin obtained from olive-oil by the degree to which a rod sank into the fatty mass in a given length of time. The rod was graduated at its upper portion, and beneath the graduations there was a disc-shaped expansion serving firstly to keep the rod steady in an outer tube through which it passed, and secondly to support a spiral spring which was adjusted in strength, so as to keep the rod at the zero point when not encountering any other resistance. Dr. R. Kissling (*Chemiker Zeitung*, 1890, 1119, and 1891, 298) has described an instrument for testing railway greases and glue. He measures the length of time taken by a rod to penetrate a certain depth of the sample, and uses several rods of fixed diameter and length, but of various weights and materials.

I have employed three different forms of apparatus for consistency tests :

Form No. 1 is really a modification of Dr. Kissling's: Instead of rods of fixed weights, which may be either too heavy or too light for a particular substance, I extend the use of a rod by having it constructed to carry additional weight if required; for this purpose the cap at the upper end has a short prolongation over which centrally perforated coin-shaped weights are slipped. This system of weighting is adopted to

avoid side strain upon the rod, experience having shown that serious error may arise from that cause.*

Form No. 2.—Measurement of the time occupied by a plummet falling a certain depth into the material, the plummet being suspended by a thread running on a pulley wheel, counterbalancing weights being carried by a small scale pan at the other end of the thread.

This form may be employed when the fluidity of the substance is too great for form No. 1; it cannot, however, be used in cases of high consistency, on account of the limited weight of the plummet taken in conjunction with its comparatively large

1.

2.

3.



sectional area. It has an advantage over the rod form in that precautions for ensuring perfect perpendicularity are avoided, if the plummet before its release is allowed to come to rest after oscillation.

Form No. 3.—Measurement of the time occupied by a disc or pan of fixed diameter in rising through a given depth of material, when drawn up by a cord variously weighted and running on a pulley wheel. This form also is restricted to compositions possessing considerable fluidity.

Weights and Dimensions.—In view of the great diversity of substances that such appliances may be required to test, and, in fact, the extreme variations observable between members of one class, or even of one substance under different conditions, it does not appear possible to give dimensions that will be generally applicable. It is better for the analyst to determine for himself, by preliminary rough tests, what will be approximately the most convenient sizes and weights for the material in hand.

* The rod glides in a perpendicular tube at the end of a horizontal arm which, like an ordinary retort ring, is adjustable at any height upon a fixed upright.

I have used a brass rod (for form No. 1) of 5 mm. diameter, 300 mm. in length, and weighing 25 grammes, the added weights being 5 grammes to 250, according to circumstances. Dr. Kissling employs rods of glass, zinc, and brass of 50, 100, and 150 grammes respectively, the diameter being 10 mm.

Comparison of Results.—It must be remembered that results obtainable by one apparatus are not readily convertible, by calculation, into terms of another; for instance, if one experimenter employ a rod of 5 mm. and another of 10, not merely will the weight in the second case be distributed over four times the sectional area, and the surface of friction (around the sides) increased fourfold as the rod descends into the material, but the length of time taken to penetrate to a certain depth will probably be greater still than is to be accounted for by these differences, inasmuch as a somewhat limited diminution of weight or increase of surface is frequently sufficient to bring the rod to a stop altogether. Then, again, a rod loses a certain amount of its weight on sinking into the material to a given depth, this loss varying with the specific gravity of the rod and of the material, and being quite different when a hollow rod weighted at the top is used, than when a rod whose weight is equally distributed throughout its length is employed.

In selecting the weights to be used, my experience leads me to believe that the best results are obtainable when the material is penetrated in a comparatively short time—20 to 100 seconds—and that by reducing the weight on a rod so as to extend the time, the accuracy is not increased.

Rules.—Unless the following rules are carefully observed, very erroneous conclusions may be arrived at:

1. The rod must descend in an absolutely perpendicular direction.
2. It must slide in its bearing with the least possible friction.
3. Conditions of temperature must be constant.
4. Vessels of one diameter must be used to contain the material under examination.
5. The rod must enter the centre of the vessel or at a fixed distance from the circumference.
6. The same depth of material must always be used.
7. The material must be allowed to rest a certain fixed time before testing.

With respect to this last rule, Dr. Kissling has suggested a period of two days, but for many reasons a shorter time is more convenient, particularly if the determination has to be made at a temperature other than that of the air at the time, or if the substance is liable to change on keeping. Of course, the maximum consistency may not be reached when a short interval is adopted, but for technical or purely comparative purposes a short period is quite admissible in many cases.

A NEW BUTTER TEST.—Whilst engaged upon tests with apparatus such as described, my attention was drawn to the possibility of distinguishing pure butter fat from that adulterated with margarine, by testing the consistency of the clarified fat after certain treatment, such as conversion to elaidin; my experiments were but commenced in this particular direction when the announcement appeared that R.

Brullé (see *Compt. Rend.*, 1893, 116, 1255) had already demonstrated the practicability of such a process. His method is as follows :

Five c.c. of the clarified fat are placed in a shallow basin 7 c.m. in diameter on an oil-bath at 148° ; when the temperature of the fat has reached 130° , a pinch of powered pumice and 8 drops of fuming nitric acid are added, the whole mixed and kept heated for twelve minutes; it is then allowed to cool to 21° C., and after one hour tested with the instrument that he terms an "oleogrammeter," which is practically identical in principle with the form No. 1 above; it consists of a movable upright rod gliding in a bearing and surmounted by a table on which weights are placed till the rod sinks rapidly into the fatty mass. Most striking differences are by this means shown between pure butter and margarine; in the former case, a weight of 250 grammes, on an average, was sustained, whilst in the latter 5,000 grammes weight was required, whilst mixtures gave intermediate figures capable of approximately indicating the amount of adulteration.*

It is scarcely necessary to remark that the actual weights borne by the fat would be largely dependent upon the shape of the basin holding it.

Whilst I am thus anticipated in this test, it is of sufficient importance to prompt me to bring it before the notice of the members of the Society of Public Analysts, and though from the recent appearance of Brullé's results I have been induced to bring forward these few notes before the conclusion of the experiments I had in view, I hope that the few words said here may lead to a more adequate treatment of the subject of *consistency* elsewhere.

ON THE DETECTION OF HEATED COTTON-SEED-OIL IN LARD.

BY WILLIAM GUSTAVUS CROOK.

(Read at the Meeting, July 18th, 1893.)

THE following is a method for detecting *heated* cotton-seed-oil in lard :

Place about ten grains of the sample to be examined in a cup-shaped porcelain capsule of about half an ounce capacity. A small disc of white filter paper (which has been soaked in hydrochloric acid, thoroughly washed with distilled water, and dried) is *just moistened* with a 12 per cent. solution of nitrate of silver, and placed in the concave part of a watch-glass, which, with the paper downward, is then inverted over the capsule containing the sample. The capsule is then put in a shallow oil-bath, to which heat is gently applied, until a thermometer in the bath reaches 240° F. The source of heat is then *immediately withdrawn*. I find that if even less than 1 per cent. of heated cotton-seed-oil be present in the sample, a very marked coloration takes place on the disc, varying from a light brown to nearly black. If the sample under examination be pure and fresh, the disc is apparently unaffected.

* Upwards of 200 samples of known composition have been examined by this process in the laboratory of the Société des Agriculteurs de France, under the strict control of a specially appointed committee. In every case the result was in accordance with the known composition of the sample.—A. H. A.

NOTE ON THE ESTIMATION OF CHLORINE IN WATER.

BY T. FAIRLEY.

(Read at the Meeting, July 18th, 1893.)

In analyzing a mineral water from Harrogate, containing only a small quantity of chlorine, a distinct difference in the amount of chlorine was observed according to the method used. In each case 500 c.c. of water was taken, and in titration with $\frac{N}{10}$ AgNO₃ solution, potassium chromate was employed as indicator. Thus the chlorine stated in grains per gallon was:

1.34 by weighing as silver chloride.

1.44 by titration of the water concentrated to one-tenth.

1.66 by titration of the water not concentrated.

(This latter result bears out those obtained by Mr. W. G. Young and others. ANALYST, May, 1893.)

On repeating the experiments I came to the conclusion that some constituent present affected the results by titration. As this water is alkaline and contains 1.71 grains of silica in solution, and as I find that soluble alkaline silicates when present in small quantity interfere with Mohr's process for the estimation of chlorine, I am led to believe that in this case an alkaline silicate (or what is potentially the same, a solution of soluble silica in sodium carbonate) is the cause of the difference observed. The water in question has been fully analyzed, and does not contain any other substance likely to interfere with the process of titration.

Methods for the Determination of Boric Acid. A. K. Reischle. (*Zeit. anorg. Chem.*, 1893, iv. 111; through *Chem. Zeit.*)—The author has made a comparative study of various methods of determining boric acid, including the following: 1. Weighing the basic acid as boric magnesium borate and determining the magnesia therein. 2. Ascertaining the amount of decomposition of a weighed quantity of sodium carbonate by the boric acid. 3. Distillation of the boric acid as methyl borate (Gooch's method), and weighing as basic calcium borate. 4. Volumetric methods. 5. Weighing as potassium borofluoride. 6. Volatilization as ammonium borofluoride and estimation by difference.

As a result of his investigation, the author concludes that in all cases where the bases present with the boric acid can be weighed as sulphates, the last appears to be the best plan; the boric acid being volatilized as ammonium borofluoride. The same method can frequently be used for the determination of free boric acid. When this process cannot be carried out, it is advisable not to rely wholly upon the other methods, but to check whichever may be used by the fluoride treatment, determining the total bases to be deducted in any convenient way.

B. B.

THE ANALYST.

Contributions to the Chemistry of Indiarubber Surrogates. B. Henriques. (*Chem. Zeit.*, 1898, xvii. 634-638). This important paper, dealing with a subject of which very little is known, is an extension of the author's work on the analysis of rubber goods already abstracted (*THE ANALYST*, xviii. 13). Rubber surrogates consisting of fatty oils that have been treated with sulphur or sulphur chloride, are known in commerce as "factice," variously spelt "facties," "fastice," or in German "faktis." In this abstract the term surrogate will, however, be employed, as being more generally understood. Two kinds of surrogate are used, the white and the brown, which differ in composition. The former occurs in the form of a light yellow friable elastic mass of neutral reaction, and somewhat pungent oily smell. Water dissolves nothing from it, acids and alkalis have but little action upon it, and it is unaffected by most organic solvents. It is characterised by its high content of chlorine, which is nearly as considerable as the amount of sulphur it contains. This chlorine is not removed by treatment with water nor with acids, whence it is evident that it cannot be present as hydrochloric acid or a chloride, but must exist in combination with the organic matter of the surrogate. Its composition, as well as that of other similar preparations, some used commercially and others prepared by the author, is given in the following table :

	S.	Cl.	H ₂ O.	Ash.	Fatty Acids.	S in Fatty Acids.	Cl in Fatty Acids.	I Num-ber of Surr.	I Num-ber of Fatty Acids.	Acetyl Number
White surrogate A	6.4	5.0	0.85	0.8	90.45	6.12	0.83	30.9	91.3	—
" " B	6.17	5.86	1.0	6.61	73.58	6.45	0.43	31.0	91.2	—
" " C	8.25	8.88	—	—	—	8.15	—	32.6	102.3	—
Brown surrogate A	15.48	0.7	—	—	—	14.14	nil	42.0	129.0	—
" " B	17.71	0.36	—	—	—	15.20	nil	42.0	125.6	—
Linseed-oil surrogate from fresh oil ...	9.34	8.84	3.02	nil	79.6	9.88	trace	56.3	160.3	21
Linseed-oil surrogate from oxidised oil ...	4.78	4.85	0.85	nil	81.67	4.06	0.60	52.6	141.2	19.6
Rape-oil surrogate from fresh oil ...	8.28	7.62	—	nil	86.89	8.34	trace	32.5	101.5	31
Rape-oil surrogate from oxidised oil	6.59	5.95	—	nil	87.95	6.54	trace	26.9	102.8	—
Poppy-oil surrogate from oxidised oil	7.68	7.44	—	nil	74.90	8.32	—	33.6	133.3	—
Linseed and rape oil surrogate from oxidised oil ...	—	—	—	nil	—	—	—	42.8	129.2	—
Castor-oil surrogate with a minimum of S ₂ Cl ₂ ...	4.82	6.70	—	nil	85.35	5.32	0.26	35.2	136.2	—
Castor-oil surrogate with a maximum of S ₂ Cl ₂ ...	10.6	8.95	—	nil	—	—	trace	21.9	143.5	105.6
"Soluble castor-oil" surrogate from oxidised cotton-seed-oil ...	6.23	5.36	—	nil	—	6.44	trace	30.8	91.5	51.3

The determination of the sulphur in the surrogates could not be accurately effected by fusion with carbonate of soda and nitre, and the plan that had been worked out for the determination of sulphur in rubber (*THE ANALYST*, xviii., 13) was employed with success. In order to estimate the chlorine on the same portion, the process was modified by the addition of nitrate of silver to the nitric acid used to oxidise the rubber surrogate, thus fixing the chlorine as silver chloride, which was afterwards decomposed by fusion with the alkaline melt and the chlorine determined on one portion, while the sulphur was estimated on another. Volhard's method was found convenient for the former purpose. The fact that a considerable percentage of chlorine is present in these surrogates accounts for the fact that bodies of this description, consisting of sulphurized and chlorinated oils, have a low iodine-absorption, a necessary consequence of their partial saturation by a halogen. When the iodine absorption is determined with the two hours period generally used in Germany, fallacious results are obtained, as the fact that the surrogates are sparingly soluble in chloroform militates against the full absorption of iodine taking place in a short time. By dint of repeated shaking, and allowing the absorption to proceed for twelve hours, the full amount of iodine is taken up, and further contact of the oil and the Hübl-solution causes no further combination. The two commercial varieties of surrogate, A and B, gave the figures 30.9 and 31, values which are noticeably low when the high absorption of the drying oil from which the surrogate is prepared is considered. The cause of the low iodine absorption is the state of approximate saturation of the oil, as explained above. In determining the iodine absorption of substances of this description the precaution must be observed to shake the surrogate solution well with the thiosulphate solution when titrating back the excess of iodine, as free iodine is entangled in the mixture of surrogate and chloroform and obstinately retained. The behaviour of the surrogate on saponification also deserves comment. As has been indicated in previous work, the surrogate is soluble in alcoholic potash, and it is found that the chlorine is eliminated, but that the sulphur is not, so that the fatty acids prepared by decomposing the resulting soap contain nearly as much sulphur as the original surrogate. The quantity of fatty acids is, however, smaller than one would anticipate, even when regard is had to the elimination of the chlorine. It was eventually ascertained that portions of the original fatty acids are present as soluble sulphonic compounds, that can be detected by evaporating the liquor (acid with hydrochloric acid) from the separated fatty acids; when the acid becomes concentrated the solution grows turbid, and then, though not before, sulphuric acid can be recognized. Apparently, therefore, the sulphur chloride used to prepare the surrogate, acting in the presence of oxygen either derived from the air or from the breaking down of a portion of the original oil, converts a fraction of the oil into sulphonated fatty acids, similar to those which exist in turkey-red oil. The occurrence of such bodies is common to all surrogates, but the amount present varies with the mode and circumstances of preparation. The elimination of the chlorine already referred to, by saponification, has the effect of causing the iodine absorption of the fatty acids to be higher than that of the surrogate itself (see table). Another deduction from the figures given above is that the samples A and B are prepared from the same kind of raw material. It may be mentioned that both these are typical English products, that made in Germany being of the same type as C.

The course of the reaction between fatty oils and sulphur chloride, as studied by the author to throw light on the mode of preparation of surrogates, is as follows: When the oil is added to a sufficient amount of sulphur chloride, the liquids mix completely, and a vigorous reaction sets in after a few minutes, much heat being evolved, the mass boiling up, and giving off fumes of sulphur chloride mixed with a little hydrochloric acid and sulphurous acid; the whole mass speedily solidifies to an elastic light yellow substance, which is only slightly sticky, and can be ground up under the pestle. On exposure to the air the substance loses any excess of sulphur chloride and hydrochloric acid it may have retained, and becomes neutral in reaction, perfectly resembling the white surrogate of commerce. If the operation be conducted in the same way, save that the reacting bodies are dissolved in some indifferent liquid, such as carbon disulphide, the progress of the reaction is slower, but the end-product is the same, the only apparent alteration being that it is somewhat porous from the evaporation of the solvent, which leaves it open in texture. If insufficient sulphur chloride be used the reaction is less violent, and a sticky mass results, which will not completely solidify even on prolonged standing. The quantity of sulphur necessary for effecting the solidification of an oil varies greatly with the nature of the oil, but is fairly constant for the same oil, as is shown by the following results:

Oil.	Per Cent. of S_2Cl_2			Per Cent. of S_2Cl_2		
	Insufficient to Solidify.			Sufficient to Solidify.		
Linseed-oil	25	...	30
Poppy-oil	30	...	35
Rape-oil	20	...	25
Cotton-seed-oil	40	...	45
Olive-oil	20	...	25
Castor-oil	18	..	20

From these figures it is plain that the drying tendency of an oil has no relation to the minimum quantity of sulphur chloride necessary to form a solid surrogate. Feebly-drying rape-oil requires less than strongly-drying linseed-oil, and castor-oil surpasses all the others in the small amount of sulphur chloride which it needs to convert it into a solid surrogate. All these surrogates are very sparingly soluble in carbon disulphide, such solubility as has been alleged being chiefly due to the presence of imperfectly converted oil, which is extracted by that solvent. The determination of the requisite quantities of sulphur chloride for the solidification of these various oils having been effected, various experimental surrogates were prepared and analyzed by the same methods as those adopted for the commercial products, and the results recorded in the table already given. None of the products thus obtained gave numbers similar to those characteristic of A and B, but the sample prepared from rape-oil much resembled that marked C, wherefore it appears that rape-oil is the raw material of this commercial variety of surrogate. One of the best means of judging the identity of these surrogates is that afforded by the comparison of their iodine numbers, and in this case the figures for this constant are very nearly identical. As C is a representative sample of white surrogate of German manufacture, it is a fair deduction that most German products of this class are prepared by the treatment of rape-oil. Further investigation was necessary to ascertain the nature of the samples

A and B. From the low content of sulphur and chlorine that distinguishes them from C, it seemed probable that they were prepared from an oil that needed for its solidification not more than 20 per cent. of sulphur chloride. The capacity of oxidised oils for absorbing sulphur chloride was therefore examined. Linseed-oil was heated for some hours to a temperature of 200° to 250° C. in contact with air and the product treated with sulphur chloride, of which it absorbed 15 to 18 per cent. in place of 30 per cent., the amount which it took before it had been oxidized. On raising the temperature to 250° to 300° C., the oxidation proceeded so far that no more than 10 per cent. of sulphur chloride sufficed to yield a solid product. This quantity corresponds with a content of 4.78 per cent. of sulphur and 4.85 per cent. of chlorine in the surrogate resulting. Other oils capable of taking up oxygen when heated, such as poppy-oil and rape-oil, behave similarly, although the percentage of sulphur chloride for their conversion cannot be reduced to the low limit attainable with linseed-oil. The power of absorbing oxygen is not the only property of an oil that influences the amount of sulphur chloride which it can take up. The nature of the glycerides of which the oil is composed is an important factor, as is evidenced by the remarkable figure for castor-oil given in the last table. The reaction between castor-oil and sulphur chloride is so vigorous that it has to be moderated by the use of an indifferent solvent in the manner described above. The surrogates prepared from oxidized linseed-oil were different from samples A and B, being darker in colour, a drawback that could, however, be avoided by conducting the preliminary oxidation at a lower temperature, a process which is now actually in use for the production of pale "boiled" linseed-oil. But the difference is more profound than can be accounted for by a variation in the mode of oxidation, as can be seen by a comparison of the figures in the first table. After some further investigation the author was able to determine the nature of the oil from which these two commercial samples had been prepared. There is an oil known in the trade as "soluble castor-oil," "oxidized castor-oil," and by similar titles, which is of English make, and is used as a lubricating oil. It is in reality blown cotton-seed-oil. This oil unites with 20 per cent. of sulphur chloride to form a solid surrogate, while unoxidized cotton-seed-oil requires about 50 per cent. of the reagent. By preparing surrogate from this material, the substance whose analysis is given at the end of the first table was obtained, and its substantial identity with samples A and B determined. The reasons for the use of this surrogate, rather than the others of German make, are no doubt that sulphur chloride is dearer than cotton-seed-oil, and economy in the former is advantageous, while it is probable that its comparative poverty in sulphur and chlorine makes it a better rubber substitute than the surrogates rich in those elements. In most cases, with the information now at our disposal, the determination of the percentage of sulphur and chlorine in a surrogate and of the iodine number of its fatty acids suffices to indicate the kind of oil from which it has been prepared. In order to ascertain whether it is possible to prepare a surrogate which is saturated with the sulphur and chlorine of sulphur chloride, the author treated castor-oil with a large excess (50 per cent.) of sulphur chloride, and extracted the mass with carbon disulphide, which removed any unchanged oil. The analytical figures given by this substance are recorded in the first table. It will be seen that the iodine absorption has not been

brought to a vanishing point, an indication that complete saturation was not attained. By further purification with glacial acetic acid and aqueous caustic soda the iodine number was reduced to 14, but could not be brought to 0. It is not quite certain from this persistent absorption of iodine that the substance still contains unsaturated compounds, as a portion of the halogen may be used up in attacking the sulphur. It is, however, evident that the main action of the sulphur chloride is the conversion of unsaturated bodies into saturated substances, and that unsaturated compounds are regenerated on saponification, as can be realized by comparing the iodine absorption of the original surrogate with that of the fatty acids obtained from it. Generally, the iodine absorption of the fatty acids is nearly equal to that of the oil before it has been treated with sulphur chloride, but castor-oil presents a noteworthy exception to this rule, the iodine number for the fatty acids from the treated oil being greatly higher than that for the untreated oil (83 to 85). This is to be explained by the supposition that the fatty acids recovered from most surrogates are derivatives of the oleic series, while those from castor-oil belong to the oxy-linoleic series $C_nH_{2n-4}O_2$. Additional light on the constitution of these bodies is thrown by their acetyl number. The usual method for determining the acetyl number was not applicable in this instance, as the liquid was too dark coloured to permit of accurate titration with phenolphthalein as an indicator. The modification adopted by the author consisted in decomposing the acetylated acids with caustic potash and distilling with sulphuric acid, the acetic acid being volatilized and determined in the distillate. The plan in the author's hands gives identical results with Benedikt's. Using this process, the number 105.6 was found for the treated castor-oil already spoken of (see table), a figure considerably lower than that given by untreated castor-oil. The deduction from this experiment is that the saponification of the surrogates does not involve the introduction of an hydroxyl group, and that the group (SH) is also absent.

The foregoing work deals with the white surrogates, and a few words must be said concerning the brown variety. The latter are dark-coloured, somewhat sticky, and contain much sulphur and but little chlorine; their price is lower than that of the white surrogates; it is probable that they are made by heating the oil with sulphur, instead of sulphur chloride. These brown products are completely saponified by alcoholic soda, and on liberating the fatty acids from the resulting soap a little sulphuretted hydrogen is given off, while the amount of sulphur in the fatty acids is but slightly less than that in the original surrogate. The iodine absorption of these surrogates is high, whence it appears that they are made from linseed-oil or mixtures of linseed and rape oil.

B. B.

The Analysis of Rubber Goods. R. Henriques. (*Chem. Zeit.*, 1883, xvii. 707-709.)—This work is a continuation of that of the preceding abstract. The author has already shown that there are two kinds of commercial rubber substitutes: the white, which is manufactured by treating various oils with sulphur chloride; and the brown, in the production of which sulphur itself is used; and that the former can be discriminated from the latter by the presence of chlorine. The quantity of chlorine is not, however, that which might be expected from the percentage known to exist

in the surrogate. Thus, two rubbers containing surrogate, the chlorine in which amounted to 3.7 and 0.9 per cent. respectively, reckoned on the mixture of rubber and surrogate, were found to contain only 0.50 and 0.37 per cent. of chlorine. Apparently, therefore, a large part of the chlorine is eliminated in the vulcanizing, being volatilized either as sulphur chloride or hydrochloric acid. The next point of analytical importance is the detection of fatty oils in the presence of surrogates which have been prepared either by treatment with sulphur chloride or with sulphur. Fatty oils are not appreciably sulphurized during the vulcanizing process, and, as the fatty acids obtained by acidifying the extract obtained by digesting the rubber under examination with alcoholic soda only contain sulphur when they have been derived from sulphurized oil, the percentage of sulphur present gives a measure of the amount derived from fatty oils in contradistinction to that produced from the saponification of any sulphurized surrogate. This view of the matter was put to the test of experiment in the following way: Rape-oil was heated with excess of flowers of sulphur to a temperature of 130° to 135° C. for several hours. A considerable quantity of sulphur dissolved in the oil and crystallized out on cooling. The fatty acids from the rape-oil thus treated were found to contain 0.98 per cent. of sulphur, thus proving that the amount of sulphur taken up by rape-oil at the highest temperature used in vulcanizing rubber is quite small, and could not lead to its confusion with surrogate. It is possible, therefore, to detect any two of the three additions to rubber—viz., white surrogate, brown surrogate, and fatty oil—and to estimate the quantity approximately; but when all three are present (a rare occurrence) the task cannot be satisfactorily accomplished.

The detection of white surrogate depends greatly on the fact that it contains chlorine, while rubber is generally free from this element. An exception, however, occurs in the case of rubber vulcanized by the cold process. Cold vulcanized rubber, so-called "patent rubber," is prepared by treatment with a solution of sulphur chloride in carbon disulphide, and might be expected to contain chlorine. It may be remarked parenthetically that an erroneous idea of what is meant by the technical term "patent rubber" is prevalent and should be corrected. The definition is sometimes found in technical works, that patent rubber is that which has been treated with caustic alkali after vulcanizing to remove the excess of sulphur. This is entirely a mistake. A much nearer approach to a definition would be that patent rubber is prepared from patent plate rubber. Patent plate rubber is not moulded or rolled, but cut from a block in manner similar to the preparation of wood veneer. In order to cut the blocks properly, it was formerly customary to bury them for some months in the earth, but now the necessary increase of firmness and consistency is obtained by artificial cold instead of the natural low temperature of the earth. A characteristic property of patent rubber is its finely-ribbed surface, which can be seen on the black rubber tubing used in the laboratory. The existence of this marking is not, however, a proof that the rubber is genuine patent rubber, as the plan has been adopted of imparting a ribbed surface to inferior rubber, prepared by rolling into sheets by pressing it against a textile material suitably rugose. The main question to be settled, namely, whether patent rubber contains chlorine or not, and whether, therefore, the methods of detecting surrogate that had been devised for

rubber vulcanized in the ordinary way are applicable to it, was investigated in the following manner :

The author vulcanized thin sheets of Para rubber by the cold process, and submitted them to analysis. Sample I. was over-vulcanized, and on that account hard and somewhat inelastic, while Sample II. was under-vulcanized and a little sticky at 100° C. The method of analysis was that described in the author's previous paper (THE ANALYST, vol. xviii. 113) :

	I.	II.
Ash of raw rubber	0.46%	0.46%
Sulphur	5.19	0.50
Chlorine	5.61	0.57
Extract with alcoholic soda	2.9	2.2
Ash of extracted residue	0.67	0.9

The total quantity of material soluble in alcoholic soda is small, and, further, contains appreciable amounts of sulphur and chlorine, whence it is apparent that rubber vulcanized by the cold process behaves towards alcoholic soda (the best solvent for surrogate) much in the same way as does rubber vulcanized by heating with sulphur. This view was confirmed by the analysis of an authentic sample of patent rubber and of the raw rubber from which it had been made :

	Patent Rubber.	
	Unvulcanized.	Vulcanized.
Ash	0.18%	0.18%
Sulphur	—	1.07
Chlorine	—	0.89
Extract with alcoholic soda	1.94	1.66
S in soda extract (calc. on original rubber)	—	0.57
Cl in do. (do. do.)	—	0.55
Rubber in do. (do. do.)	1.94	0.54

Similar results were expected in ordinary commercial samples of patent rubber, as it is repeatedly stated in technological handbooks that only pure Para rubber can be cut into patent plate. The first sample was one of cheap quality from a North German factory, the two others being samples of good rubber tube of different origin :

	I.	II.	III.
Ash	0.67%	— %	0.2%
Sulphur	3.68	2.37	1.86
Chlorine	3.63	2.51	1.33
Residue after extraction with alcoholic soda	84.3	92.1	94.5
Containing :			
Ash	1.8	—	0.88
Sulphur	1.92	1.77	0.83
Chlorine	1.31	1.54	1.17
Extracted by alcoholic soda :			
Sulphur and chlorine	4.08	1.57	1.19
Organic matter	12.5	6.5	5

In all cases the percentages are reckoned on the original rubber.

In order to ascertain the nature of the surrogate that had been added to Sample I., and had been detected by the foregoing results, a large quantity of the

rubber was extracted with alcoholic soda, the alkaline extract acidulated and the fatty acids removed by solution in ether. They constituted a thick oily residue which gave the following analytical figures :

Sulphur	6.29%
Chlorine... ..	0.35
Iodine absorption	92

These numbers are almost identical with those previously obtained for white surrogate, and it therefore appears that "patent rubber" occurs in commerce adulterated with vulcanized cottonized or rape-oil. This conclusion, deduced from the figures obtained by analysis, was confirmed by a communicative manufacturer, according to whom patent rubber has been made for a number of years of mixtures containing as much as one-third surrogate, although the practice has not been generally known to exist. The fact that a rubber is sold as "patent rubber" is no longer to be considered as a guarantee of genuineness, even when the mode of preparation is that proper to patent rubber.

B. B.

Hydrolysis of Fats by Concentrated Sulphuric Acid. A. Prager and J. Stern. (*Chem. Zeit.*, 1893, xvii. 880.)—By using sulphuric acid of the exact strength recommended by Kreis (91.53 per cent. H_2SO_4) in his latest communication (*ANALYST*, vol. xviii. 145), the authors have certainly obtained numbers agreeing better with the Reichert-Meissl numbers than those which they obtained when they followed Kreis' original specification, but *only with pure butter*. A mixture of butter with margarine yields numbers by Kreis' method which are higher than the Reichert-Meissl numbers in proportion to the percentage of margarine present. It is generally admitted that the Reichert-Meissl method will not detect 10 per cent. of margarine in butter with certainty; much more true is this of the Kreis method. The following figures may be quoted :

		No. of c.c. of $\frac{N}{10}$ Alkali required by 5 Grammes of Pure Butter.					
		I.	II.	III.	IV.	V.	VI.
Reichert-Meissl	...	27.23	30.78	31.79	30.14	30.88	30.15
Kreis	27.84	29.96	32.28	31.18	31.22	31.12

MIXTURE OF BUTTER WITH MARGARINE.

Percentage of Margarine.	No. of c.c. of $\frac{N}{10}$ Alkali required by 5 Grammes of the Sample.	
	Reichert-Meissl.	Kreis.
0	26.05	26.63
10	23.64	24.50
20	21.14	24.53
40	16.27	19.35
60	11.38	16.09
100	0.85	0.98

In the majority of cases the numbers obtained by Pinette's modification were nearly as much lower than those obtained by the Reichert-Meissl method as the Kreis numbers are higher, although in several instances (not here quoted) the numbers agreed fairly well.

Percentage of Margarine.		No. of c.c. of N_{10} Reichert-Meißl.	Alkali required by 5 Grammes of the Sample.		
0	...	30.14	Pinette.	...	Prager and Stern.
10	...	27.36	28.78	...	30.32
20	...	24.64	25.48	...	27.40
40	...	19.10	23.02	...	24.58
60	...	13.44	17.35	...	19.16
100	...	2.65	11.21	...	13.56
			1.96	...	2.70

The authors conclude that removal of sulphurous acid cannot be omitted, and must be effected by the method proposed by them. A. G. B.

The Determination of Manganese by Means of Hydrogen Peroxide.

A. Carnot. (*Compt. Rend.*, 1893, cxvi. 1375; through *Chem. Zeit.*)—In order to determine manganese by means of the available oxygen of its peroxide, it is necessary in the first place to prepare a peroxide of definite composition. This can be effected in several ways.

In the first method the compound containing manganese is dissolved in strong nitric acid, with the addition of small quantities of potassium chlorate, the manganese being thus converted into MnO_2 . The separation of manganese from other metals is complete, save in the case of iron, which accompanies the manganese to some extent, and can be separated by solution of the peroxide in nitric acid with the addition of hydrogen peroxide, evaporation, and reprecipitation with strong nitric acid and potassium chlorate. After thorough washing of the purified manganese peroxide first with cold and finally with hot water, it is determined by measuring the oxygen evolved by treating it with hydrogen peroxide and nitric acid in the manner described below.

Better results than those yielded by the chlorate method are obtained by the use of ammoniacal hydrogen peroxide to bring the manganese to a definite state of oxidation. Hydrogen peroxide in excess is added to the solution containing the manganese, excess of ammonia quickly added, and the mixture boiled for some minutes, whereby a dark-brown precipitate of the composition Mn_2O_{11} is produced. Large quantities (10 to 20 grammes) of ammonium salts, notably, the acetate, hinder the formation of this peroxide, but the difficulty can be overcome by the use of a large excess of hydrogen peroxide. Should the precipitation be effected in the presence of copper, zinc, nickel, or cobalt, the manganese peroxide contains a small amount of the oxides of these metals, which can be removed by several precipitations. Small quantities of iron do not interfere, but when iron predominates a preliminary separation with nitric acid and potassium chlorate is advisable.

The final determination of the manganese is carried out by placing the peroxide, however obtained, in a flask, together with dilute nitric acid, running in a known volume of hydrogen peroxide, and measuring the air displaced by the oxygen evolved.

B. B.

NOTE BY ABTRACTOR.—The method is probably feasible, as it has been used by the undersigned for red lead; but as the manganese peroxide is obtained pure, it would be simpler to ignite and weigh as Mn_2O_4 .

B. B.

The Adulteration of Margarine with Sunflower Oil. A. Jolles and E. Wild. (*Chem. Zeit.*, 1893, xvii. 879.)—Two samples of margarine recently examined by the authors appeared to be adulterated with cotton-seed oil on account of their high iodine absorption and the positive indications given with Becchi's solution and the nitric acid test. A third sample of the same class gave the following figures:

Melting-point of fatty acids	43° C.
Solidifying-point	"	"	...	39° C.
Iodine absorption	60.34
Becchi's test	Positive indication
Nitric acid test	Negative indication.

On account of the failure to obtain any characteristic brown colour on shaking with nitric acid (sp. gr. 1.37), the authors considered that some vegetable oil other than cotton-seed oil was present. Such an oil, to be remuneratively used as an adulterant, must be of pleasant flavour and not much dearer than cotton-seed oil, these conditions excluding olive, rape, and linseed oil. Seeing that the iodine absorption of sunflower oil is about 127 and that of cotton-seed oil 107, the figure given above for the suspected sample corresponds with an adulteration of 22 per cent. of the latter and 15 per cent. of the former, whence it follows that cotton-seed oil could not have been present, as it would have been indicated by the melting and solidifying point of the fatty acids, by the elaidin test and by the brown coloration with nitric acid. Pursuing their investigations, the authors found that the difference of behaviour of cotton-seed oil and sunflower oil with nitric acid (of sp. gr. 1.37)—the former giving the brown coloration above mentioned, and the latter showing no change of tint—serves as a criterion even when as little as 10 per cent. of either is present in margarine. A trial mixture containing 85 per cent. of margarine and 15 per cent. of sunflower oil had an iodine absorption of 61.5 per cent., the iodine absorptions of its constituents being 47 and 127 respectively, while a mixture of 90 per cent. of margarine and 10 per cent. of sunflower oil gave the number 55.37, and one of 85 per cent. of margarine and 15 per cent. of cotton-seed oil 55.8. The first result, therefore, corresponds with that of the adulterated sample. The nitric acid test was also effective in distinguishing between these synthetic samples. This test can be conveniently made on a chloroformic solution of the oil.

It is noteworthy that the price of cotton-seed oil has recently risen, so that it is now nearly double that of sunflower oil—a circumstance that gives ground for suspecting the extended application of the latter as an adulterant. B. B.

The Nessler Reaction for Ammonia. B. Neumann. (*Chem. Zeit.*, 1893, xvii. 880.)—L. L. de Koninck has stated (*Zeit. anal. Chem.*, 1893, xxxii. 188) that Nessler solution gives no reaction with ammonia in alcoholic solution. The author has found, on the contrary, that, in alcohol of strength ranging from 1 to 100 per cent., a trace of ammonia gives a definite reaction with Nessler solution, and, in fact, that the influence of the alcohol is almost negligible. He attributes the discrepancy to some want of sensitiveness in the specimen of Nessler solution used by De Koninck.

B. B.

The Examination of Beeswax. G. Buchner. (*Chem. Zeit.*, 1893, xvii. 918, 919).—The best method of examining beeswax is that due to Hübl; but there are many fictitious waxes (e.g. such a mixture as 35 parts of stearic acid, 165 parts of japan wax, and 300 parts of paraffin wax) which can be added to beeswax without being detected by this plan, or the melting point, or specific gravity. The author's method of examination is as follows.

The wax is first melted with distilled water, and the resulting cake of wax allowed to cool, removed and dried. Should the water be acid to litmus—which may arise from the bleaching of the wax with acid, followed by imperfect washing—the cake is remelted with water several times, as the presence of acid may affect the determination of the constants of the wax. The final fused product must be clear. The purified wax is examined in the conventional manner.

I. *The wax gives normal figures* (acid number, 19-21; ester number, 73-76; saponification number, 92 to 97; ratio of acid to ester number, 3.60 to 3.84). It may be either genuine wax or a cleverly sophisticated sample. Further tests are therefore necessary.

(1) Stearic acid is looked for by Döttger's modification of Fehling's test. One grm. of wax is boiled for some minutes with 10 c.c. of alcohol, cooled to 18-20° C., filtered, the filtrate treated with water and shaken. Stearic acid separates in flocks and collects on the surface of the liquid, while the underlying aqueous portion becomes clear. One per cent. of stearic acid can be thus detected. Should as much as 7-8 per cent. of stearic acid be present, it remains diffused through the water as a thick creamy mass.

(2) Rosin is looked for by Donath's method, modified by E. Schmidt. Five grms. of wax are treated with 4 to 5 times as much nitric acid of sp. gr. 1.32 to 1.33, and heated therewith to boiling for 1 minute. An equal volume of water is then added, and afterwards ammonia in excess. The liquid is poured off the separated wax, and is yellow at most should the wax be pure, but more or less brown should rosin be present. One per cent. of rosin can thus be detected. A blank test should be made with pure wax.

(3) Glycerides (e.g., japan wax and tallow) are recognized by the isolation of glycerine. For this purpose the residue (still warm) left after the execution of the Hübl method of examination (*v.s.*) is evaporated on the water-bath; the alcohol driven off, water added, the filtrate concentrated and tested for glycerine by heating with potassium bisulphate.

Should these tests give negative results the wax is genuine, seeing that the acid number and other constants are normal. It may be noted that the commonest adulterants are paraffin wax, japan wax, carnauba wax, and stearic acid.

II. *The wax gives abnormal figures.*—In this case the sample can be condemned out of hand, save when the only abnormality is a somewhat higher acid number, and tests for rosin and stearic acid give negative results. The acid number in bleached wax may be as high as 24 without the sample being necessarily sophisticated; otherwise adulteration has certainly been effected. The nature of the adulterant is ascertained by the tests under I., and the quantity calculated from Hübl's constants. In special cases the glycerine present must be determined, and the paraffin wax by

Buirine's method. The author draws attention to the fact that although but little pure beeswax is sold, yet the stuff on the market gives nearly normal figures, a proof that the fraudulent application of chemical knowledge is frequent. B. B.

REVIEWS.

ANALYSIS OF MILK AND MILK PRODUCTS. H. LEFFMANN and W. BEAM. Philadelphia: Blakiston, Son, and Co. 1893. 12mo., 92 pp.

Drs. Leffmann and Beam have recognised the want of a manual on milk analysis and have produced what must be pronounced the best book on the subject in the English language; for its size it is marvellously complete, and well up to date, work appearing in the month of publication having been noticed.

On page 10 the authors state that "caseinogen" is frequently used to designate the form in which casein exists before coagulation; Halliburton, however, applies this term to the proteid whether in solution or precipitated by acids, and reserves the name casein for the curd produced by rennet; lower down a starch-converting enzyme is said to occur in milk, but Béchamp's "galactozymase" from cow's milk is described by him as only liquefying starch paste, and not possessing hydrolytic action; in human milk he describes a true starch-converting enzyme.

The analyses of different kinds of milk on page 11 do not seem quite correct; there is evidently a clerical error in sow's milk; Besana, who has contributed most to the chemistry of ewe's milk, gives a higher percentage of fat than 6·8 per cent.; the proteids of cow's and goat's milk are rather high, and those of human milk rather low as averages.

We are glad to see it recorded that the growth of certain organisms as *B. cholerae* is hampered by the natural organisms of milk.

D'Hout's figure for the fat in skimmed milk (0·02 per cent.) is certainly too low; the authors use "skimmed" as synonymous with "separated."

On page 18, Babcock's method of total solid estimation is erroneously attributed to Richmond.

We are somewhat surprised that on page 19 the quantitative estimation of sulphates should be recommended as a means of detecting watering; few waters contain as much as 0·01 per cent. SO_3 (10 parts per 100,000), and more than that quantity has been found in milk.

The printer does not always manage to place the decimal-point correctly on page 20, where the error due to the extract from Schleicher and Schüll's coils is given as 0·003 per cent. in place of 0·03 per cent.

It is hardly correct to say that Hehner and Richmond's formula was based on perfect processes of fat-extraction, as they used unextracted coils, and made a correction; by a fortunate compensation of errors this formula is near correctness, as shown by many hundreds of analyses. Richmond's slightly-modified formula, which is more scientifically correct, does not seem to be generally known.

A very useful modification of Sebelein's method of casein and albumin estimation is described on page 34.

At the bottom of page 37 it is directed to dissolve mercury in an equal weight of nitric acid in the preparation of Wiley's acid mercuric nitrate; Wiley's directions are to use twice the weight of nitric acid. Vieth's formula for correcting the polarization figures for the volume of the fat is not given.

On page 54 a table is given of the limits adopted in various countries; public analysts will open their eyes at seeing their limit looking miserably small at the bottom of the list.

The chapter on butter is perhaps the most unsatisfactory part of the work, the only method described being the Reichert process; and although this may serve in America, where fine mixtures do not flood the market as in England, it does not alone suffice for the needs of English analysts, who have to grapple with small adulterations.

Probably Drs. Leffmann and Beam have considered methods of distinguishing between proteic, amidic, and ammoniacal nitrogen in cheese as beyond the scope of their book; these are, however, of importance, when the quality of a cheese, apart from its purity, has to be determined.

We would advise every analyst, teacher, and student of milk analysis to obtain this book; it should be widely used as a handbook on the subject, as although it is not free from imperfections, these are mild beside the glaring inaccuracies of the present day text-books. We are inclined to think that the American spelling will somewhat militate against its sale in this country.

H. D. R.

MODERN MICROSCOPY: A HANDBOOK FOR BEGINNERS. By M. I. CROSS and MARTIN J. COLE. London: Baillière, Tindall and Cox. Price 2s. 6d.

This work contains much useful information on the choice of a microscope, and will be found of great value to persons who are about to make a selection of such an instrument. The first portion of the book, written by Mr. Cross, is devoted to descriptions (accompanied with excellent illustrations) of several of the instruments and their accessories now before the public, beginning with the simpler forms and proceeding to the more complex. By the aid of these the novice is able to ascertain the form of instrument best suited to his requirements. Plain and simple directions are given for the use of the instrument, and special reference is made to the important subject of illumination, the various condensers being illustrated and their use described. In the second part, which is from the pen of Mr. Cole, who is an old and experienced hand, instructions for cutting, staining, and preparing tissues for examination are succinctly given. These, if carefully followed, cannot fail to lead to the attainment of good results. The work, which is clearly printed and neatly bound, is exceedingly moderate in price.

W. J. S.

EXPERIMENTS UPON MAGNESIA ALBA, QUICK-LIME AND OTHER ALKALINE SUBSTANCES. By JOSEPH BLACK, M.A. Edinburgh: W. F. Clay.

This little book, which emanates from the Alembic Club of Edinburgh, is a reprint of a paper of Dr. Black's, first published in 1755, copies of which are now scarce and difficult to obtain. As stated in the preface, the objects in reprinting the paper are: "(1) To enable students of the history of chemistry to possess themselves of a copy of this important contribution to the foundation of chemistry as an exact

science; and (2) to place within the reach of every student of chemistry a model of clear reasoning and of inductive investigation, which is second in this respect to nothing in chemical literature that has appeared in much more recent times." In spelling and punctuation the original has been closely followed. It is to be followed by reprints of the important writings of other British chemists. W. J. S.

CORRESPONDENCE.

To the Editors of THE ANALYST.

DEAR SIRS,—It was a wish to avoid controversial matter that led us to state "Stokes and Bodmer's method is suitable, but probably not very reliable" (ANALYST, xviii. 171); we thought that, as we had methods at our disposal that were more satisfactory, it was unnecessary to detail our reasons for thus dismissing it. As Messrs Stokes and Bodmer now take exception to the statement, and accuse us of condemning the Pavy method of titration without trial, merely because Hehner had criticized it adversely, we deem it advisable to give the reasons which we had previously omitted.

We did consider Hehner's results, showing the influence of carbonates and chlorides, as having a direct bearing on the subject; chlorides to small amount being introduced with the sugar solution, and carbonates being formed by absorption of carbon dioxide from the air by the caustic alkali, both before and after making the solution.

Chittenden (*Studies from the Lab. of Physiol. Chem., Yale Univ.*, ii. 46) and Ogúda (*Jahr. f. Thierchemie*, xv. 275) have also shown that Pavy was misled by the use of this method.

We, however, recognized that by taking the precaution of standardizing the solution on pure milk and cane sugar (inverted) under the conditions of the experiment, these theoretical objections might be overcome; an examination of the results given by Messrs. Stokes and Bodmer, in their original paper, where they say that these precautions were taken, convinced us that the method was "probably not very reliable" (for condensed milks).

In that paper the authors give six determinations of known amounts of cane-sugar added to milk; the amounts added were small, from 1.0 per cent. to 10.5 per cent., and are consequently not comparable directly with the quantity contained in condensed milk. We have, therefore, calculated the error per cent. on the amount taken:

No.	Taken.	Found.	Error Per Cent.
1	1.00	0.95	- 5.0
2	3.25	3.19	- 1.9
3	2.90	2.82	- 2.8
4	3.20	3.05	- 4.7
5	5.50	5.68	+ 3.3
6	10.50	10.80	+ 2.9
		Probable error	± 3.6

Assuming that condensed milk contains an average of 40 per cent. of cane-sugar, the probable error in the determination of this method would be $\pm \frac{3.6 \times 40}{100}$, or ± 1.44 per cent.

The limit of accuracy of a method is about 2.7 times the probable error, and therefore the Stokes-Bodmer method of estimating cane-sugar in condensed milk is reliable to 4 per cent.

Being in possession of methods giving greater accuracy than this, we did not feel justified in devoting our time to a critical examination of Pavy's method of titration; we wish it to be distinctly understood that our criticism was not intended to apply to cases in which the percentage of sugar is small, as it is well known that the method of titration with the substance to be estimated increases in accuracy inversely to the amount of substance in a nearly hyperbolic ratio.

After the detailed reasons we have given we do not see how Messrs. Stokes and Bodmer can object to our very qualified condemnation of their method in reference to condensed milk analysis as "probably not very reliable."—Yours, etc.,

H. DROOP RICHMOND.
L. K. BOBELEY.

THE ANALYST.

OCTOBER, 1893.

THE LATE MR. C. W. HEATON, F.I.C., F.C.S.

It is with much regret that we chronicle the death, at the age of 58, of Mr. C. W. Heaton, the treasurer for many years past of the Society of Public Analysts. Mr. Heaton was best known as Professor of Chemistry in the Medical School of Charing Cross Hospital, a post which he filled from the year 1862 until the time of his decease. He also held the post of Examiner in Chemistry to the Royal College of Physicians and the Royal College of Veterinary Surgeons, and was Public Analyst to the parish of St Martin-in-the-Fields. His chief work in applied chemistry was connected with the subject of coal-gas and its purification, also with the recovery of sulphur from waste products; but he directed no inconsiderable attention to the subject of food analysis, and contributed various papers to the pages of *THE ANALYST*. As a pupil of the late Mr. Dugald Campbell, and subsequently as an assistant to the late Dr. Stenhouse, Mr. Heaton was one of the best-known links between the earlier and later generations of consulting chemists, and his friendships in the profession were many and far-extending. His loss will be mourned not merely among his colleagues in the Society, but in the whole circle of chemical life throughout this country.

ON SOME MODIFICATIONS IN THE BABCOCK MACHINE.

By CHAS. R. C. TICHBORNE, LL.D., F.I.C., D.P.H., etc.

(Read at the Meeting, July 18th, 1893.)

ALTHOUGH a great deal has been written upon the value of the Babcock, Leffmann-Beam, and Lactocrite machines, perhaps I shall be excused if I say a few words as regards my own experience of the results obtained with the Babcock, as specially contrasted with the ordinary ether analysis. This experience has been gained after some thousands of estimations.

I may sum up my opinion of the proper place of the Babcock as only being a valuable adjunct to a busy laboratory, and to be worked under the supervision of a trained expert. The working of such machines by a trained hand is a very important element of success. I say this advisedly, because many of the Dairy Co-operative Societies with whom I am connected fail to get the value out of machines like the Lactocrite or Babcock.

My observations and experience confirms what has already, in many cases, been ably pointed out by members of the Society of Public Analysts, but as some of them are not quite agreed, I will enumerate my own conclusions without going into details.

1. Any machine working by centrifugal force for the separation of butter fat is largely dependent upon the mechanical condition of the fluids, and is liable to hidden errors. It therefore should never be used by the Public Analyst for prosecuting purposes.

2. If the separated butter line is clear from curd and above the average, it may be looked upon as a reliable assay. If anything below 3 per cent., it should be looked upon with caution, and centrifugal instruments are unreliable for milk containing less than 2 per cent. of butter-fat.

"Why do I select the Babcock machine?" is a question which has been partially answered by others, notably Mr. Embrey. My chief object is to work with an apparatus which gives me butter-fat alone, without the use of such objectionable adjuncts as fusel-oil. Again, the high speed required in the other instruments is a very serious objection when they are in constant use.

It has been pointed out that a correction is required, which Mr. Embrey views as a constant quantity, and fixes at 0.3. I have come to the same conclusion as regards a correction being necessary, but my experience does not lead me to think it is a constant quantity, but that it must be changed according to the richness of the milk in the machine, *i.e.*, that the correction for milk under 2 per cent. of butter-fat will be slightly different from that about 3 per cent. I should mention that according to the modification I am about to detail in the machine, the reading of the butter lines is conducted at a much more uniform temperature than was the case with the old machine.* The fact probably enables one to watch the question more effectively.

The strength of the acid, as shown by previous observers, is of great importance, and the success of the estimations mainly depends upon this point: either too strong or too weak is equally objectionable.

In the old machine we were directed to put hot water into the tank. The following are the directions:

"As soon as the bottles have been sufficiently whirled they should be filled to the neck with hot water. The cover should then be replaced and the machine turned for one or two minutes, after which more hot water is added, filling the tube to about the 7 per cent. mark.

"The fat will slowly rise into the graduated tube, losing its cloudy appearance as it passes through the hot water. When all the bottles are filled the cover is put upon the tank and the machine again turned for a short time.

"During this time the water in the tank should be kept hot by pouring in a quantity of boiling hot water before starting the machine. If the fat in some of the tubes still has a cloudy appearance, the cover should be placed upon the tank and more boiling water added for a few minutes, when the fat should become clear and in condition to be measured. The clearing may be hastened by whirling the tubes while hot.

"The fat when measured should be warm enough to flow readily, so that the line between the acid liquid and the butter-fat will quickly assume the horizontal position

* As every operator has his own method of working, and as the machines may differ to some slight extent, I have thought it better not to give any arbitrary correction. As a matter of fact my experience points to an addition of 0.3 to quantities less than 2 per cent., 0.2 on 2.5 per cent., and 0.1 or 0.0 above this percentage.

when the bottle is removed from the machine. Any temperature between 110° F. and 150° F. will answer, but the *higher* temperature is to be preferred.

"The slight difference in the volume of fat due to this difference in temperature is not sufficient to materially affect results. A difference in temperature of 40° F. will make less than $\frac{1}{10}$ th per cent. difference in milk containing 5 per cent. of fat."

It will be seen that this process entailed a considerable amount of work to get a nice clear separation, and it is only in the summer that we can work with any degree of certainty, commensurate with quickness, and the value of the machine is entirely dependent upon the perfect separation of the fat in a clear condition. This clouding of the fat is most objectionable in the winter time with a laboratory at a temperature of 50° F. The "hot water" was chilled on the first two or three revolutions; the revolving case was simply blowing cold air across the *graduated stem*, this being the part of all others which required to be in a perfectly warm and uniform condition, so that the fat should remain permanently liquid during the whirling of the bottles. This is remedied in the new form of Babcock by putting a pint of boiling water into the lid of the chamber, which is made hollow for the purpose. The heat is thus applied to the stem, not to the body of the bottles. Any cool air that is whirled in strikes upon the hot lid and is whirled across the thin stems.

The old and present machines are provided with a case or chamber, in which the bottle charged with milk and acid are put, in sockets arranged round the chamber. The narrow, elongated stems, in which the butter-fat collects after whirling, and is measured, are placed close to the lid of the chamber, like the spokes of a wheel. In the old machine this is simply a tin cover. In the new machine it is a chamber about an inch deep and covering the whole top of the machine. It is, of course, removable for the purpose of getting out the bottles. At the apex there is a little funnel, through which a pint of water at 212° F. is introduced.

Now, it must not be forgotten that the final separation between the butter-fat and the acid and insoluble nitrogenous matter takes place in the stems and not in the bodies of the bottles, and thus the importance of keeping these parts of the apparatus above the melting-point of butter-fat.

The following is the result of comparative working of the instruments :

					New Machine.	Old Machine.
1.	2.3	2.2
2.	3.6	3.5
3.	3.2	3.0

The great saving, however, is in time, the operation being perfectly completed in two terms of five minutes duration each. A quantity (say a pint) of boiling water having been placed in the lid, the assay is effected at a uniform temperature, which is of considerable importance where the milk is rich in butter-fat, as the error due to contraction or expansion is exaggerated. The expansion is stated in the original to make no difference. This is not correct. I find that it holds good for 2 per cent. of fat, but that if there is 3 or 4 per cent. of butter-fat this error becomes much more important. With the same ratios it would read that the difference of temperature which makes 2 per cent. of fat read $2\frac{1}{10}$ per cent., would in the case of 4 per cent. of fat make an error of 0.2 per cent., or the 4 per cent. would read 4.2 per cent.

Mr. Lister, whose machines are described in the May number of *THE ANALYST*, has adopted our lid on his machines. I may as well state that the form of machine before you has been protected by myself and Mr. Thompson, of Messrs. Carson and Sons, Dublin, who made the first one at my suggestion.

VINEGAR.

BY A. H. ALLEN AND C. G. MOOR.

(*Read at the Dublin Meeting, July 18th, 1893.*)

RECENT events, well known to the members of this society, have compelled public analysts to give more attention to the chemistry and manufacture of vinegar than the majority of them had previously done. In a recent number of *THE ANALYST* there appeared an abstract of a paper by us in which we gave a number of definitions of "vinegar" which we had collated from various sources. Some of these definitions were not drawn by their authors with the expectation of their being quoted in judicial proceedings, and are not unnaturally wanting in the requisite precision on the one hand and the necessary elasticity on the other. Before making an attempt ourselves to formulate a more satisfactory definition of "vinegar," we propose to review shortly the chief processes by which the various products commercially posing as vinegar are manufactured.

No naturalist has any difficulty in classifying a *cow* as an animal and a *cabbage* as a vegetable; but in the case of some of the lower forms of life the biologist is at a loss how to regard the specimen. So it is with vinegar, some commercial products almost defying classification.

The so-called vinegars of commerce may be broadly divided into two classes: those which are the product of the consecutive alcoholic and acetous fermentations, and those which are not. The view is pretty general amongst public analysts that products of the former class have alone the right to be called "vinegar," unless some adjectival prefix be used, such as "wood-vinegar."

Of course the original vinegar was made from wine, as the derivation of the word shows; but in this country wine-vinegar has been almost wholly superseded by malt-vinegar. This again has been superseded, so far as a product made from malt only is concerned; for, just as the beer-brewer has discovered that the substitution of unmalted barley for a portion of the malt is distinctly advantageous, so the vinegar-brewer invariably uses more or less unmalted grain in admixture with the malt. But the beer-brewer has, since the abolition of the duty on malt, largely replaced the malted and unmalted barley by maize and rice, or the "glucose" prepared therefrom by the action of dilute acid; and the vinegar-manufacturer has not been slow to follow suit, and to use sugar and molasses in addition. So general has this practice become that the vinegar-manufacturers who still brew from malted and unmalted barley only might be counted on the fingers of one hand (without reckoning the thumb). Under these circumstances, it is evident that the term "malt-vinegar" is a misnomer, and it behoves public analysts to consider how far they will be justified in permitting the sale of products of mixed origin under the title of "malt-vinegar." There is undoubtedly a popular preference for, or prejudice in favour of,

malt-vinegar; but while manufacturers are quite ready to pander to this preference, whether it be justified or not, they do not always supply an article to which the name "malt-vinegar" can be properly applied, even when used in the extended sense of a product brewed from a mixture of malted and unmalted grain. The contention that by such a term a vinegar merely *flavoured* with malt is to be understood will scarcely bear investigation. Take the case of jam. Are we to understand by "raspberry-jam" an article composed chiefly of apples or turnips, and containing only enough raspberry to give it the characteristic flavour? Or under the title of "malt-whisky" are we to admit a spirit made not only from unmalted barley, but also from rice, maize, dari, potatoes, sugar, molasses, etc.? It would undoubtedly prevent much heartburning, and greatly simplify the question at issue, if the term "malt-vinegar" were definitely abolished.

But the substitution of other fermentable materials for barley is not the only trade-practice the legitimacy of which calls for consideration. The albuminoids present in malt-wort and other vegetable infusions have a prejudicial effect on the keeping properties of the vinegar produced therefrom, especially in the case of the weaker qualities, and it is the object of the scientific vinegar-manufacturer to reduce these to a minimum. Hence it is the practice of some manufacturers to distil a portion of their product—which, contrary to the general impression, can be done very cheaply—reserve the stronger portion of the distillate for sale as "distilled vinegar," and add the weaker fractions to some of the undistilled article. Distilled malt-vinegar is not, as is sometimes erroneously assumed, merely dilute acetic acid. On the contrary, it contains quite appreciable quantities of alcohol, acetal, acetic ether (and probably other esters), furfurol, aldehyde, etc.; and has a highly characteristic taste and odour which appear to be greatly appreciated in Scotland, where distilled vinegar is seen more frequently than the coloured article.

We have obtained the following figures by the analysis of distilled *vinegar* (not mere acetic acid) manufactured by some of the leading vinegar-brewers:

	A.	B.	C.
Specific gravity	1.0077	1.0080	1.0055
Acetic Acid (grammes per 100 c.c.)	5.16	5.80	3.88
Total solids " "	0.006	0.013	0.032
Ash " "	None.	None.	None.

It is evident that the addition of either of the above distilled vinegars to an undistilled product would tend to alter materially the proportions of certain of the constituents of most value to the analyst in judging of the origin and mode of manufacture of a sample of vinegar. In fact, the manufacturer who practises the addition of a distilled vinegar to his undistilled product runs considerable risk of having his article certified to contain added acetic acid.

Two samples of still-residues from the manufacture of distilled vinegar have been examined by us. They were brown, and of the consistence of treacle. On analysis they yielded the following figures:

	A.		B.	
Total solids	43.20	per cent.	72.30	per cent.
Ash	8.88	"	9.21	"
Alkalinity of Ash (K_2O)	2.07	"	1.40	"
Phosphoric Acid	2.67	"	0.24	"
Nitrogen	2.63	"	2.63	"

A was a residue from a true barley vinegar. B was derived from a vinegar brewed with a considerable proportion of rice.

Under the name of "Essig Sprit," or "spirit-vinegar," there is now sold considerable quantities of an article of foreign origin, which is manufactured by subjecting previously distilled potato-spirit to the acetous fermentation. The product has many of the characters of English brewed vinegar, and of course contains those peculiar secondary products of the growth of *Mycoderma aceti* which give vinegar certain of its valued characters. It is usual to add a certain proportion of malt-wort to the spirit, or of malt-vinegar to the fermented product. *Essig Sprit*, as imported, contains about 13 per cent. of acetic acid, and a notable proportion of acetic ether; but the colouring matter is added in England, whereby the article is imported as "acetic acid," and escapes the duty payable on "vinegar." The following figures show the composition of "Essig Sprit" in its concentrated form, and of the "spirit vinegar" of commerce obtained by diluting and colouring it:

		ESSIG SPRIT.	"VINEGAR."			
			A.	B.	C.	D.
Grammes per 100 c.c.	Specific gravity ...	1.0152	1.0095	1.0086	1.0068	1.0096
	Acetic acid ...	11.26	4.68	4.50	3.75	4.80
	Total solids64	0.79	0.50	0.40	0.65
	Ash06	0.04	0.05	0.03	0.06
	Alkalinity of ash ...	—	Trace	Trace	Trace	0.01
	Phosphoric acid ...	Trace	0.002	None	None	Trace
	Nitrogen ...	0.014	0.0084	0.0098	0.0110	0.013
= Albuminoids		0.089	0.053	0.062	0.069	0.082

Another product of German manufacture, which finds its way into English commerce to a considerable extent, is prepared by fermenting potato-spirit with *Mycoderma aceti*, neutralising the dilute acetic acid so obtained with "pure lime," and distilling the resultant calcium acetate with an acid. Another manufacturer neutralises the acid with soda instead of lime. By this means a very concentrated acetic acid is obtainable, the strength of which ranges from 40 to close upon 90 per cent. When suitably coloured and flavoured, such acid is extensively sold under such names as "malt acid," "vinegar essence," "vinegar extract," etc., from which a factitious vinegar can be prepared by simple dilution with water. The following figures were obtained by us some time since by the analysis of samples of this so-called "malt acid" and the "vinegar" obtained by its dilution.

		"MALT ACID."		"VINEGAR."	
Grammes per 100 c.c.	Specific gravity ...	1.0825	1.0977	1.0191	1.0125
	Acetic acid ...	88.02	45.4	3.84	8.11 (<i>sic</i>)
	Total solids ...	2.77	12.14	2.00	0.27
	Phosphoric acid (P_2O_5)		0.017	Present.	
	Ash ...	0.15	0.180	0.04	
	Nitrogen ...		0.113		
	= Albuminoids ...		0.70		
Sulphuric acid (free)			0.74	0.063	

The production of a liquid containing more than 14 to 15 per cent. of acetic acid by the acetous fermentation is simply impossible, and the practical limit is reached in Essig Sprit with about 13 per cent. But vinegar-brewers do not, as a rule, aim at producing an article containing more than 6 per cent. at the outside. Hence the mere fact of a liquid having an acidity corresponding to 80 or even to 40 per cent. of acetic acid is absolute proof that it is not a product of fermentation, whatever the manufacturer may declare. Nor can a vinegar resulting from fermentation be, in practice, materially concentrated by evaporation, although absolute acetic acid has a boiling-point somewhat above that of water. Thus, on distilling a sample of commercial malt vinegar, containing 4.17 grammes of acetic acid per 100 c.c. to half its bulk, we found the distillate to contain 2.87 grammes of acetic acid per 100 c.c., while the liquid remaining in the retort contained 4.88 per 100 c.c. On reducing 500 c.c. of the same vinegar to 15 c.c. by distillation, without the use of any fractionating arrangement, the residual liquid contained only 10.1 per cent. of acetic acid. A similar experiment made on a mixture of malt vinegar and acetic acid B. P., containing before distillation 14.16 grammes of $C_2H_4O_2$ per 100 c.c., yielded a distillate containing 10.92 of acetic acid, while the half remaining in the retort retained 17.4 grammes of acid per 100 c.c.

We have recently met with as many as ten samples of vinegar containing a notable quantity of free sulphuric acid, though it is right to add that there is reason to believe these were all supplied to the retailers by the same "vinegar maker." This article was stated to be made by adding to the vinegar brewed from 2 cwt. of malt and 1 cwt. of molasses 25 gallons of "wood-vinegar," and sufficient water to reduce the strength of the mixture to a little over 4 per cent. of acetic acid. From these data we calculate that the product would contain about 8 per cent of true fermentation-vinegar. On analysis it has given the following figures :

Grammes per 100 c.c.	Specific gravity	1.0130 to 1.0138
	Acetic acid	4.0 ,, 4.2
	Total solids	1.6 ,, 1.7
	Ash	0.06 ,, 0.09
	Phosphoric acid	Trace.
	Sulphuric acid (free)	0.22 ,, 0.30
	Nitrogen	0.016 ,, 0.020

The residues obtained by the evaporation of these vinegars were quite black.

The solicitor who defended the vendors of this article, the casks containing which were labelled by the manufacturer "pure malt vinegar," stated that its manufacture cost as much as true malt vinegar. Taking the price of 25 per cent. acetic acid at 7s. per cwt. (as per list in the *Chemical Trade Journal*), it follows that a 4 per cent. "vinegar" could be made therefrom for a trifle over 1½d. per gallon.

We have tried to compare the taste of dilute acetic acid with that of sulphuric acid, and find the latter to leave a sourness on the palate equal to an acetic acid of fully four times the strength. Hence 0.25 per cent. of sulphuric acid is palatably equivalent to about 1.0 per cent. of acetic acid, and the manufacturer who adds it can reduce his bill for the latter by 25 per cent. Arsenic-free sulphuric acid of 1.725 specific gravity is quoted at 26s. per ton; whence it follows that a dilute acid

equal in sour taste to a vinegar containing 4 per cent. of acetic acid can be manufactured for 2d. per 100 gallons!

We may here express our opinion that the use of the term "pyroligneous acid," to express the quality of wood acetic acid used in the production of factitious vinegar, is misleading. By pyroligneous acid is commonly understood an acid obtained by the distillation of wood. By conversion into a calcium or sodium salt it can be readily purified to a much greater extent than if neutralization be omitted, and when recovered from its salt by distillation with a mineral acid it is technically known as "acetic acid." Although the term pyroligneous acid is very convenient for indicating the origin of an acetic acid, it implies the presence of tarry matters, which, in our experience, do not exist in the acetic acid employed for manufacturing factitious vinegar.

In order to obtain more reliable information respecting the composition and range of variation of vinegar prepared from different materials, we have obtained samples of representative vinegars from most of the leading vinegar-brewers, and have analysed these samples in some detail. The actual figures obtained we reserve for the present. Our analyses have included the determination of specific gravity, total acid in terms of acetic acid, total solids, ash, the alkalinity of the ash in terms of K_2O , the phosphoric acid, the nitrogen by Kjeldahl's process, free sulphuric acid when present, tests for heavy metals, and qualitative tests for alcohol, furfural, aldehyde, acetic ether, etc.

With regard to phosphoric acid, the importance of which as an indication of the origin of vinegar was pointed out in "Commercial Organic Analysis," vol. i., p. 389, and has since been insisted on by *Hehner* (*ANALYST*, xvi., 81), its value as a quantitative method is seriously discounted by the influence on the composition of the wort produced by the use of hard water. As, however, any precipitation of phosphate of calcium thus occasioned is more than counterbalanced by the formation of a corresponding soluble salt, the proportion of total ash is not reduced thereby.

The "alkalinity" of the ash when obtained is also a useful indication, but we have not found the results very concordant. A better plan is undoubtedly to determine the actual potash, as is practised by *R. R. Tatlock*. It is very difficult to obtain a fairly white ash by the combustion of vinegar-residues, and if the temperature be too high, or the ignition very prolonged, there is a serious danger of losing some of the more volatile salts. In our later experiments, therefore, we have adopted the practice of sulphating the ash. This does not interfere with the determination of potassium by direct precipitation with platonic chloride in the portion of the ash soluble in water. In practice we find it convenient to take 5 c.c. for the determination of the extract, and having weighed the total solids, to add another 20 c.c. of the same vinegar, and again evaporate. When reduced to the consistency of a syrup, a few drops of strong sulphuric acid are added, the evaporation completed, and the residue ignited at a low red heat. By these means a perfectly white and practically infusible ash is readily obtained. As a rule, 115 to 120 parts of the sulphated ash represent 100 parts of unsulphated ash, the extreme limits hitherto observed being 106 and 130. Of course the ratio will vary with the composition of the ash, and

consequently with the origin of the vinegar, and may in the future afford very valuable information.

With regard to the determination of nitrogen, we find it convenient to operate on 20 c.c. of the vinegar, though a smaller quantity will suffice. When concentrated to a syrup, we employ Gunning's modification of the Kjeldahl process. We have also obtained very good results by Stock's oxide of manganese process.

For the determination of free acetic acid in vinegar we employ 10 or 20 c.c. of the sample, which we titrate in a weighed porcelain dish with caustic soda, using phenol-phthalein as an indicator. There is not the least difficulty in noting the end of the reaction, without diluting the vinegar or taking any other precaution. The neutralized liquid is distilled, and the distillate tested for the volatile constituents of vinegar. Alcohol is readily recognised by the iodoform test, and aldehyde by the rosaniline sulphite reaction. Acetic ether can be determined by Berthelot's method, similar to Koettstorfer's process for oils. Furfural, we at first thought might be indicative of acetic acid derived from wood, especially as the ordinary acetic acid of commerce gives abundant evidence of the presence of furfural when tested by the aniline acetate reagent. Unfortunately there is either furfural or a body giving a similar reaction produced when sugar is caramelised. On treating the resulting caramel with water, distilling, and testing the distillate with the aniline acetate reagent, a strong furfural reaction is obtained.

We have attempted to find some compound which would be directly indicative of a brewed vinegar as distinguished from the various forms of distilled acetic acid. Alcohol is only practically valuable for this purpose in special cases, and the objections to relying implicitly on the natural presumption from its presence are obvious. Glycerin, another constant product of the alcoholic fermentation, is very difficult to determine in the presence of some of the other constituents of vinegar. We have attempted, therefore, to determine the succinic acid, which substance the experiments of Pasteur showed to be a constant product of the alcoholic fermentation. There are great difficulties in its accurate determination under the conditions with which analysts have to deal in practice, but our experiments in this direction are not complete, and we are not hopeless that we may be able to devise a practicable method of dealing with it.

In interpreting the results obtained by the analysis of vinegar, it is important to eliminate the variations due to differences of dilution. This may be effected to some extent by expressing all the items per 100 parts of acetic acid, but a far preferable plan is to adopt the principle suggested by *Hehner (ANALYST, xvi., 82)*, who calculates the amount of phosphoric acid, ash, etc., per 100 parts of the original solids of the vinegar. These are deduced from the fact that 180 parts of glucose yield theoretically 120 parts (or two-thirds of its weight) of acetic acid. Hence, by multiplying the acetic acid found in the vinegar by 1.5, and adding to the product the percentage of total solids still existing in the vinegar, the "original solids" are obtained. In practice, the actual yield of acetic acid is rarely more than two-thirds of the theoretical amount, so that a more accurate expression of the original solids of the wort would be obtained by multiplying the percentage of acetic acid by $\frac{9}{4}$ ($= 2.25$), and adding the extract to the product.

We purposely leave any discussion of the deductions from the foregoing analytical data for the present, as also the actual figures we have obtained by the analysis of the numerous samples of typical vinegar, with which we have been kindly furnished by a number of the leading vinegar-brewers of the kingdom. We may, however, conveniently give here the results of the analysis of two special vinegars. One of these was manufactured on the large scale from locust-beans; the other was a domestic product, obtained simply by fermenting a solution of brown sugar with brewers' yeast, and allowing the liquid to stand in a loosely-covered vessel:

		Locust-bean Vinegar.		Sugar Vinegar.	
Grammes per 100 c.c.	Specific gravity	...	1.0217	...	1.0284
	Acetic acid	...	7.26	...	6.90
	Total solids	...	2.47	...	4.69
	Ash	...	0.31	...	0.46
	Alkalinity (K_2O)	...	0.11	...	—
	Phosphoric acid	...	trace	...	trace
	Nitrogen	...	0.03	...	0.046
	= Albuminoids	...	0.189	...	2.290

SOMERSET HOUSE AND WATER ANALYSIS.

By F. W. STODDART.

THE following notes may be of interest, as throwing some light upon the views entertained by the Somerset House chemists upon this very important subject.

The water of a shallow well supplying a poor district in Bristol was found to yield very marked evidence of sewage pollution of the "oxidized" type common in similar situations, and action was taken by the Sanitary Authority to close the well. Analyses put forward on behalf of the owners of the property agreed fairly in all respects with my own, except that nitrates were confessedly ignored as being of no importance; and, following the lines of a well-known text-book, the water was described as of excellent quality.

In view of considerable opposition, an independent opinion was considered advisable, and Mr. Hehner was requested to report on the water. His figures are included in the annexed table, and his opinion, based on those figures, coincided with that I have already expressed, viz., that the water was polluted with sewage, and dangerous to health.

The well in question is situated at the foot of a millstone grit hill, composed of beds of rock of the usual character, dipping sharply at an angle of about 25° . It is sunk entirely in this formation, is 45 feet deep, and is not puddled, but built round with loose stone, uncemented. It is immediately surrounded by a nest of houses of a poor class, and is, indeed, placed beneath a small paved yard attached to one of the houses, the other end of the yard being occupied by a privy. Numerous drains, some of which have recently been defective, are known to exist within a few feet of the well, and there is an old graveyard at a distance of a few yards. The steep slope of the hill, and the whole of the table land at its summit, are thickly inhabited, forming, indeed, a great part of Clifton; so that there is no possible gathering ground of a satisfactory description.

The natural unpolluted water from this formation almost always yields less than 0·1 grain per gallon of nitrogen as nitrates, with total solids ranging from 20 to 30 grains. The chlorine is variable according to the position, but samples from the immediate neighbourhood have contained 1 to 1·5 grains.

The hearing of the case was very prolonged, and ultimately the bench decided to refer the matter to Somerset House. A sample was sent on June 4, and after a fortnight's delay a series of questions as to the circumstances of the well was received; as the replies were necessarily in every instance unfavourable to the water, it was naturally supposed that any doubts as to its polluted character would be dispelled.

No report, however, was forthcoming, and only after repeated applications for some expression of opinion was a letter received containing a proposal that the matter should *stand over until November*.

As the local Authority was intent on setting its house in order in view of a possible cholera invasion, and as the evidence of pollution was quite conclusive, this proposal was naturally declined; and finally, after further pressing, the following report, dated July 25, was received on August 9, a period of more than two months having elapsed since the sample was despatched. It will be noted that Dr. Bell's name, which headed the list of signatures in the previous correspondence, does not appear in connection with the report, the figures of which are included for convenience in the table.

"Laboratory, Somerset House, W.C.

"Judging from the figures shown under the terms 'albuminoid ammonia' and 'oxygen absorbed,' the water is regarded as of moderately good quality, and affords no evidence of serious contamination with organic impurities. The proportion of nitrates is high, but they are not in themselves injurious, and do not necessarily indicate that the water has been exposed to recent sewage contamination. There are good grounds for believing, from the character and amount of the chief mineral matters contained in the water, that the great bulk of the water is derived from a deep or distant source, and that the well is not dependent for supplies from the soil of the immediate neighbourhood; for it would be reasonable to expect more definite evidence of present sewage contamination if leakage took place from the drain-pipes which lie—it must be admitted—in almost dangerous proximity to the well.

"Although this water is unsatisfactory in some respects, and we should not recommend it, we do not feel justified from a consideration of the results of the analysis in giving an opinion that the use of the water for potable purposes would be dangerous to health.

"As witness our hands this 25th day of July, 1893.

"(Signed) R. BANNISTER, F.I.C., F.C.S.
G. LEWIN, F.I.C."

Little comment is needed on my part. The undecided tone unfortunately characteristic of certificates from the same source is here displayed to perfection.

The suggestion that, because in the opinion of Messrs. Bannister and Lewin the water is not wholly supplied from the polluted subsoil, it is therefore fit for drinking is, perhaps, especially worthy of note.

It is, however, simply lamentable that this water, a typical example of the class most insidiously dangerous, because inoffensive or even attractive to the unaided senses, should be described as safe by an official department naturally of importance in the eyes of the general public, thereby raising a serious obstacle to the most important of all sanitary improvements.

There are large numbers of shallow wells in Bristol, and probably in many other large towns, supplied more or less completely from a subsoil of the filthiest description, which furnish water of this character; and this applies especially to those districts in which the cholera raged most furiously in previous epidemics.

There is, of course, no necessity whatever to make Somerset House a court of reference in matters relating to water supplies, and I imagine no public health officer acquainted with the details of this case will consent to such a procedure in future.

	May 18.	May 20.	June 4.
	Stoddart.	Hegner.	Somerset House.
Saline ammonia	0.0017	0.0008	0.002
Albuminoid ammonia	0.003	0.0022	0.0047
Nitrogen as nitrates	1.79	1.81	2.44
Chlorine	3.70	3.71	3.58
Oxygen absorbed 15 min.	0.018	0.015	—
" " 4 hrs.	0.025	0.043	0.026 (3 hrs.)
Total dissolved solids	53.0	53.6	55.5
Phosphoric acid	Strong trace	Very heavy trace	—
Sulphuric acid	—	7.92	—
Loss on ignition	—	6.16	—
Total hardness	36.0	—	28.1
Permanent do.	21.0	—	—

All results in grains per gallon.

Sheep's Milk and the Manufacture of Cheese therefrom. C. Besana. (*Staz. Sper. Ag. Ital.*, xxiii. 572.)—The author has made a study of the milk of the sheep and the products derived therefrom, a research undertaken at the instance of the Minister of Agriculture. Some preliminary results have already been published in the *Bollettino di notizie agrarie*, 1887, 57; 1890, 26; and 1891, 18. Much of this important memoir is not suitable for an analytical journal, but the following portions will be of interest to analysts:

The Milk of the Sheep.—A sheep under ordinary circumstances has been found by the author to yield 45 to 50 litres of milk per annum; Pouriau has found with the Roquefort flock 55 litres a year (*La Laiterie*, 1881, 12), and Fleischmann gives as limits 25 to 140 litres (*Das Molkereiwesen*, 1876, 88); he quotes some Dutch sheep which gave 500 (?) litres a year. This quantity varies greatly in individuals,

and according to the season; thus, with three sheep kept for special observation, the following mean daily quantities were found:

No.			Litres per Day.			Meals counted and measured.
1	0.374	41
2	0.552	35
3	0.275	15

In his observations at Prima Porta in 1891, after a prolonged drought, the author found that 1,000 sheep gave 66 litres per meal, as against 150 and 130 litres per meal found in former years after normal seasons; the richness of the milk was also much less in 1891 (*see post*).

Density of the Milk.—The density of the milk of the three sheep mentioned varied from 1.037 to 1.043, with an average of 1.0395 (176 observations). The variations of the mixed milk of a flock were 1.035 to 1.039; Fleischmann (*loc. cit.*) gives 1.035 to 1.041 as the limits.

Influence of Temperature on the Density of Sheep's Milk.—The author has calculated a table for correcting the densities of sheep's milk for temperature:

Temperature.		Difference in Degrees for 1 Degree Cent.		Correction.
5° to 10°	...	0.20	...	Subtract 1.25 and 0.20 for 1 degree under 10°.
11° to 15°	...	0.25	...	Subtract 0.25 for each degree under 15°.
16° to 20°	...	0.30	...	Add 0.30 for each degree over 15°.
21° to 25°	...	0.32	...	Add 1.5 and 0.32 for each degree over 20°.
26° to 30°	...	0.35	...	Add 3.1 and 0.35 for each degree over 25°.
31° to 35°	...	0.37	...	Add 4.85 and 0.37 for each degree over 30°.
36° to 40°	...	0.40	...	Add 6.7 and 0.40 for each degree over 35°.

Effect of Watering on the Density.—The mixed milk of two sheep was diluted with varying amounts of water:

Sample.		Density.		Diminution.		Diminution for each 10%.
Genuine	...	39	...	—	...	—
5%	...	37	...	2	...	—
10%	...	35½	...	3½	...	3½
20%	...	32½	...	6½	...	3
30%	...	30	...	9	...	2½
40%	...	28	...	11	...	2
50%	...	26	...	13	...	2

Fat Globules.—The microscopic appearance of sheep's milk is like that of the cow; the globules are, however, larger in size. The author found the globules of cow's milk to vary from 0.0016 mm. to 0.0100 mm., and of sheep's milk from 0.0047 mm. to 0.0309 m.m. This character may be of service in cases of adulteration of cow's milk with sheep's milk.

Creamometry.—Sheep's milk throws up no cream in 48 hours, owing to its great density and viscosity; if diluted with an equal bulk of water, 14 per cent. of cream was thrown up in 24 hours and 20 per cent. in 48 hours; after 48 hours the cream-layer did not increase.

Chemical Composition.—The analysis of eight samples are given:

- A. Milk of a flock of 2,700 sheep at Santa Maria di Galera, 1887, evening.
- B. *Ibid.*, morning.
- C. Milk of a flock of 2,500 at same place, 1890, evening.
- D. *Ibid.*, morning.
- E. Milk of a flock of 2,498 at Prima Porta, 1891, evening.
- F. *Ibid.*, morning. There had been a very bad season this year, and the yield was very low (*see ante*).
- G. Milk of a flock of 2,000 at Rome, 1892, morning.
- H. Milk of three sheep kept for experiments at Lodi, 1892, morning.

	A.	B.	C.	D.	E.	F.	G.	H.
Water ...	78.37	79.04	77.27	77.92	80.15	80.83	78.56	78.37
Fat ...	8.99	8.90	10.38	10.04	7.25	7.12	9.41	9.30
Proteids ...	6.55	6.10	6.28	6.22	6.03	5.84	6.14	6.20
Sugar ...	5.08	5.04	4.98	4.93	5.20	5.20	4.93	4.99
Ash ...	1.01	0.99	1.09	0.89	1.37	1.01	0.98	1.09
Total ...	100.03	100.13	100.00	100.00	100.00	100.00	99.96	100.02
Density ...	1.0381	1.0374	1.0381	1.0379	1.0375	1.0373	1.0362	1.0391

The author prefers to neglect Samples E and F in calculating the mean composition, as they are undoubtedly abnormal; the mean composition of the others is:

Water...	78.23
Fat	9.50
Proteids	6.26
Sugar	5.00
Ash	1.01
Total	100.00
Density	1.0378

The most important difference between this milk and cow's milk is the large amount of fat in sheep's milk—about two and a half times that in the milk of the cow; the amount of proteids is also much greater.

The extraordinary richness in fat gives this milk a greasy feeling when touched, a character also possessed by all its products; when the milk is warmed to the temperatures employed in cheese-making, oily drops separate.

Determination of Fat.—The author finds that the use of Marchand's lacto-butyrrometer gives results sufficiently exact for practical dairy work; he finds it necessary to dilute the milk with an equal volume of water and to add a few drops of ammonia. It is advisable to make three experiments, using respectively two, three, and four drops of ammonia, and to take the highest result as the correct one. In the whey obtained from sheep's milk the fat can be directly estimated by this method.

Spontaneous Acidification.—Sheep's milk takes about twice as long to curdle spontaneously as cow's milk. The author does not attribute this wholly to differences in composition, but thinks that as the sheep were kept in the open country, while the cows live in sheds, the sanitary conditions are more favourable in the case of the sheep. This character is, however, very precious for cheese-making.

Action of Rennet.—The curd yielded by sheep's milk is dense, homogeneous and firmer than that yielded by cow's milk. The milk of the sheep requires about double the quantity of rennet necessary for that of the cow. This holds good whether ordinary rennet, purified rennet, rennet extract, or 'rennet flower' (*Cynara cardunculus*) is used to curdle the milk.

Butter from Sheep's Milk.—The only practical method of obtaining the cream for churning is by the use of a separator. A Laval Baby separator was used by the author, the rate of running being 50 litres an hour. A sample of milk of density 1.035, containing 10.25 per cent. of fat, gave a skim-milk of density 1.047, containing 0.40 per cent. of fat, 96 per cent. of the fat being removed. In churning it is necessary to work at a temperature below 10° C., or a satisfactory consistency will not be obtained. From whey the cream may be separated as clotted cream, by setting, or by a separator; the latter is preferable.

Characters of Sheep's Butter.—The colour, unless it has been coloured, is nearly white; it has a much more greasy feeling than cow's butter, and softens at a lower temperature, though its melting-point is the same—33.5° to 35°, as against 33° to 37.7° for cow's butter. A sample contained 11.4 per cent. of water. The Reichert-Wollny figures of two samples were 27.6° and 26.5°.

Rennet.—The author finds considerable difference between ordinary rennet (obtained direct from the stomachs) and purified rennet (obtained from the former by precipitation with sodium chloride). By the use of the latter the cheese obtained was free from the peculiar odour of the sheep, which was always produced by the action of ordinary rennet, and which has always been supposed to be a characteristic of sheep's cheese. The cheese obtained by purified rennet was indistinguishable from ordinary cheese.

The remainder of the paper is devoted to the details of the preparation of various kinds of cheese, and is devoid of analytical information.

H. D. R.

On the Determination of Sand Fraudulently or Accidentally Mixed with Seeds. G. Cugini. (*Staz. Sper. Ag. Ital.*, xxiii. 568).—The author shakes 5 to 20 grammes of the seeds to be tested with chloroform in a separating cylinder, and weighs the portion which sinks to the bottom.

H. D. R.

The Determination of Chromium in Ferro-Chromium. J. Spüller and S. Kalman. (*Chem. Zeit.*, 1893, xvii. 880, 881.)—Finely-powdered ferro-chromium is rapidly attacked by fusion with a mixture of three parts of caustic soda and one part of sodium peroxide (which is now an article of commerce). The chromium can be conveniently determined in the watery extract of the melt (after the decomposition of the excess of sodium peroxide) by treatment with ammonio-ferrous

sulphate and backward titration with potassium permanganate. The same fusion mixture serves for the qualitative recognition of chromium and manganese. Silver vessels should be used, as platinum is badly attacked. The use of a mixture of caustic soda and sodium peroxide is preferable to that of sodium peroxide alone, as the latter acts too violently on oxidisable alloys, such as ferro-chromium, ferro-silicon, etc. B. B.

NOTE BY ABTRACTOR.—The authors give no prescription for getting rid of the excess of sodium peroxide previous to the volumetric estimation of the chromium present as chromate. It could probably be effected by acidifying with acetic or sulphuric acid, and running in dilute potassium permanganate until a faint pink colour appeared, due to the completion of the decomposition by the permanganate of the hydrogen peroxide resulting from the action of the acid on the sodium peroxide. B. B.

The Determination of Chromium in Ferro-Chromium. J. Spüller and S. Kalman. (*Chem. Zeit.*, 1893, xvii. 1207, 1208.)—The authors find that a mixture of caustic soda and sodium peroxide is a good flux for ferrochromium and similar refractory substances. (See preceding Abstract.) Their experiments, which were carried out with two samples of ferrochromium nominally containing 40 per cent. and 60 per cent. of chromium respectively, led them to adopt the following method. 0.35 grms. of the very finely divided alloy is fused in a silver dish with 4 grms. of pure powdered sodium peroxide and 8 grms. of pure powdered caustic soda, the ore and flux being intimately mixed with a silver spatula, and the heating conducted cautiously over a smoky flame.* During the first five minutes the edge of the charge begins to fuse, and fusion of the whole is complete after a further period of ten minutes. Heating is continued for an hour, during the last quarter of which the melt is stirred with a silver spatula. Sudden heating at the beginning is not advisable. The basin is allowed to cool to 40-50° C., and placed bodily in hot water and the melt extracted. The basin appears to be considerably attacked, as the average loss for each time of using was 4 to 5 centigrammes on a dish weighing 38 grms. The watery solution of the melt is yellow or red-brown when the sample is poor in manganese, and it contains sodium ferrate as well as chromate. When manganese is present in considerable quantity, the solution is blue or green, from the coloration due to sodium manganate. Only traces of sodium peroxide can usually be recognised in the aqueous solution, as it decomposes when treated with hot water, especially in the presence of sodium ferrate and manganate. This property is taken advantage of to reduce these substances without reducing the chromate. 0.3 to 0.6 gm. of sodium peroxide is added little by little to the solution of the melt, until its colour appears pure yellow, from the presence of sodium chromate only. The excess of sodium peroxide is best destroyed by passing CO₂ into the hot solution for an hour, and continuing the heating for another quarter of an hour. (The author recommends the use of a bath of graphite instead of a sand bath, presumably on account of its higher conductivity.) By this process the caustic

* The smokiness of the flame cannot be essential, as the melt is an oxidising one; an argand burner turned fairly low would probably prove suitable.—B. B.

soda and sodium peroxide are converted into sodium carbonate with the liberation of hydrogen peroxide from the latter; the hydrogen peroxide, being unstable in alkaline solution, is dissipated on heating, and the only highly-oxidised substance remaining is sodium chromate.

The aqueous solution of the melt, after having been thus purified, is transferred to a 500 c.c. flask without previous filtration, made up to the mark and a portion filtered from the ferric oxide, manganese dioxide, and alumina that remain as insoluble matter. 100 c.c. of the filtrate is used for the determination of chromium by Schwarz's method. The measured quantity is diluted with a litre of cold boiled distilled water, and acidulated with 20 c.c. of sulphuric acid containing 1 part by volume of H_2SO_4 to 5 parts of water; 100 c.c. of a solution of ammonio-ferrous sulphate containing 14 grms. of the salt per litre are added, with constant stirring, and the residual ferrous iron titrated with permanganate solution of such strength that 1 c.c. = 0.00423 grms. Fe. The strength of the ammonio-ferrous sulphate is then determined by diluting it with water as in the actual determination of chromium acidulating and titrating with permanganate to the same intensity of colour as before.

A gravimetric process, in which the same flux is used, is also described. The alloy is attacked precisely as previously detailed; the melt extracted with water, and freed from sodium ferrate and manganate and excess of sodium peroxide by the methods already laid down. The insoluble matter is then filtered, and washed with a solution of sodium carbonate to prevent its passing through the filter. The filtrate contains sodium silicate and aluminate as well as chromate, and is, therefore, taken down to dryness after the addition of 1.5 grms. of ammonium nitrate, and the residue dissolved and filtered. The filtrate is acidulated with hydrochloric acid, heated on the graphite bath, and the chromic acid reduced by 1 gramme of pure potassium nitrite. The resulting solution containing chromium as chromic oxide is precipitated hot with ammonia, avoiding an excess. After long standing at 90°C. , the precipitate is filtered off, washed until chlorides cannot be detected in the washings, dried and ignited, together with the filter-paper, in a platinum crucible. A little chromium chromate is left with the silica and alumina in the residue after the evaporation with ammonium nitrate. The residue is, therefore, treated with hot dilute hydrochloric acid, whereby all the chromium and the alumina are dissolved. The solution is precipitated with dilute ammonia, and the precipitate filtered, washed, ignited, and fused with caustic soda and sodium peroxide as before, and the alumina and chromium separated by evaporation with ammonium nitrate in the manner detailed above. The small quantity of chromic oxide finally obtained is ignited and added to the main precipitate. This total chromic oxide is only an approximate measure of the content of chromium of the alloy, as, having been precipitated in the presence of alkaline salts, it not only contains alkalis, but some chromate due to oxidation of a portion of the chromic oxide in contact with a powerful base. It is, therefore, placed in a porcelain dish, covered with hot water, and rubbed down with a thick glass rod, the solution filtered, and the residue repeatedly washed. The filtrate, which contains alkaline chromate, is transferred to a porcelain basin, acidulated with hydrochloric acid, reduced with a few drops of a solution of potassium nitrite, and

precipitated with ammonia, the chromic oxide being collected, ignited, and weighed, and its weight added to that of the main precipitate which has undergone this process of purification.

The analytical figures given are in the case of the nominal 40 per cent. ferrochromium, 43.95 and 44.0 per cent. by the volumetric method, and 44.20 per cent. by the gravimetric process; the corresponding figures for the nominal 60 per cent. alloy are 57.60 to 57.68 per cent. and 57.92 per cent. The slightly higher results of the gravimetric process are to be attributed to the difficulty of removing traces of silica and alumina from the chromic oxide.

The reliability of the method of fusion was investigated by examining the insoluble matter for residual chromium; none was found.

The mixture of caustic soda and sodium peroxide can be advantageously used for many analytical purposes, where at present a mixture of sodium carbonate or caustic soda and potassium nitrate or chlorate is commonly employed. The powerful oxidizing properties of sodium peroxide, and the ease with which an excess may be removed, make it a valuable reagent. Iron pyrites can be oxidized in the same manner as ferrochromium, and the determination of sulphur affected without evaporation to get rid of nitric acid, as in the case where a mixture of sodium carbonate and potassium nitrate is used as a flux. Two samples of chrome iron ore, one containing much magnesia from Greece, the other a pure specimen from Bosnia, were completely opened up in $1\frac{1}{2}$ hours. Commercial chromic oxide, so-called "chrome-green," can be completely attacked in three quarters of an hour, and apparently the process should be applicable to the determination of chromium in chromium steel. The authors are engaged in investigating this point.

B. B.

The Determination of Boron. H. Moissan. (*Compt. Rend.*, 1893, cxvi. 1087, through *Chem. Zeit.*)—Gooch's method, which consists in the volatilization of boron in the form of methyl borate, gives the best results. The author's modifications of procedure are as follows. The distilling apparatus consists of a bulb flask with a side tubulus, like that of a fractional distillation flask, but sloping first up and then down. A tap funnel is ground into the neck of the flask, and reaches down into the bulb. The flask is heated in a calcium chloride bath. The tubulus of the flask is connected with a vertical spiral condenser, and opens into a small conical flask immersed in very cold water and serving as a receiver. The connection of the end of the condenser with the receiver is made by means of a two-holed cork, the second hole being provided with a tube bent at right angles and trapped by a bulb apparatus containing ammonia (1 : 5), which catches any trace of boric acid that has escaped the spiral condenser and the cooled receiver.

In conducting an analysis the boron is first converted into boric acid, which is generally best effected by heating it with nitric acid in a sealed tube. The resulting solution, which should be of as small bulk as possible, is distilled to dryness. (In the case of an already formed borate, 1 c.c. of nitric acid is added before distillation.) The flask is then removed from the bath, 10 c.c. of methyl alcohol added, and the contents again distilled, the operation being repeated four times with fresh portions

of methyl alcohol; 1 c.c. of water and 1 c.c. of acid are then added, and distillation again carried out, three further treatments with methyl alcohol being requisite. The complete volatilization of the boron is proved by allowing a drop of the last distillate to fall on a strip of paper, which is then burnt, and the characteristic green colour due to boric acid looked for. Should any boric acid be detected, further distillation is requisite. The use of a calcium chloride bath in the distillation is to heat the walls of the flask to a temperature above the boiling-point of methyl alcohol, and thus avoid bumping. The contents of the receiver and bulb tube are emptied into a platinum crucible containing pure slaked lime which has been previously weighed as caustic lime, the crucible being meanwhile surrounded with ice-water to moderate the reaction should much acid be present. After the lapse of a quarter of an hour to allow of the completion of the reaction, the contents of the crucible is tested to make certain that it is alkaline, evaporated on the water-bath at about 70° C. to dryness, and the temperature gradually raised, ignition being completed over the blowpipe first with the crucible covered, then open. It is allowed to cool in a desiccator over caustic lime, is weighed and reheated until its weight is constant. The gain of weight is the B_2O_3 obtained. The requisite lime is prepared in the crucible in which it is to be used. Calcium nitrate is ignited until it is partially decomposed, in which state it can be conveniently stored. When needed a portion is ignited to constant weight, and used as described above. For the retention of 0.5 grm. of B_2O_3 , 8 to 10 grms. of lime are requisite. The method, when carefully performed, is accurate and generally applicable. B. B.

NOTE BY ABTRACTOR.—It is futile to attempt to render '8 to 10 grms.' of lime completely caustic over any ordinary blowpipe. The use of a muffle is indispensable. B. B.

Titration of Alkaline Liquids Containing Hypochlorite. C. Ullmann. (*Chem. Zeit.*, 1893, xvii. 1207, 1208.)—It is frequently necessary in examining liquids that have been subjected to electrolysis to determine their alkalinity when chlorine and hypochlorites are present. For example, when a solution of sodium chloride, which has been electrolyzed, and contains (in addition to undecomposed sodium chloride) sodium hydrate, sodium hypochlorite, and possibly sodium chlorate, is to be tested, the usual methods are not available. Ordinary indicators are destroyed, and such acids as hydrochloric, nitric, sulphuric and oxalic are inapplicable. The author has found succinic acid to be of service and uses it in the following manner. A measured quantity of the liquid to be examined is heated with an excess of a standard solution of succinic acid until the smell of hypochlorous acid is no longer recognised. Phenol-phthalein is then added, and the solution titrated back with caustic soda. The utility of the succinic acid results from the facts that it is not volatile, that it decomposes carbonates and hypochlorites, but not chlorides, and that it is not affected by the oxidising and chlorinating action of the electrolyzed salt solution. B. B.

Quantitative Precipitation in Presence of Hydroxylamine. P. Jannasch and J. Mai. (*Berichte*, 1893, xxvi. 1786.)—As an example of this application of

hydroxylamine the authors quote the following method for precipitating chromium hydroxide from bichromate. About one gramme of potassium bichromate is dissolved in 50 c.c. of water and heated with 10 c.c. of concentrated hydrochloric acid and 25-30 c.c. of alcohol, until the liquid is green and the greater part of the excess of alcohol has been expelled. The solution is diluted to 300 c.c., and heated; 2 grammes of pure hydroxylamine hydrochloride are added and the chromium precipitated by a considerable excess of ammonia. The violet-red coloured precipitate is immediately filtered through two filters, washed with boiling water, and dried sufficiently to enable the filter to be transferred to a platinum crucible, where it is ignited. The excess of ammonia used has no solvent action on the precipitate, which filters more readily, is more easily washed, and is less liable to contain silica than is the precipitate with ammonia obtained in the usual way.

A. G. B.

A New Method for the Determination of Yellow Phosphorus. J. Toth. (*Chem. Zeit.*, 1893, xvii. 1244, 1245.)—The following method has been devised for the determination of yellow phosphorus in mixtures containing phosphoric acid as a product of oxidation of the element. The principle underlying it consists of the extraction of the phosphorus with CS_2 , precipitation of the dissolved phosphorus as silver phosphide, oxidation of the latter with nitric acid and determination of the resulting phosphoric acid in any convenient way.

The accuracy of the process thus outlined was ascertained as follows: A small piece of yellow phosphorus was cut out of the centre of a stick, quickly immersed in alcohol, then in ether, and finally in a vessel containing olive-oil, which had been proved to yield no phosphoric acid when shaken with nitric acid. The olive-oil, together with the vessel containing it, was weighed before and after the introduction of the phosphorus, and thus the weight of the latter ascertained. CS_2 , which had been proved free from H_2S by its remaining colourless when shaken with silver nitrate solution, was then added, dissolving the phosphorus, and the resulting solution was diluted with a large quantity of olive-oil, so that each gramme of oil contained 1-2 mgrm. of P. Weighed portions of the oil were taken to test the method of estimation. For example, 20 grammes of the oil were shaken with 10 c.c. of a 5 per cent. solution of silver nitrate, and 10 c.c. of water in a flask of 200-300 c.c. capacity until the separation of silver phosphide was complete. 20 c.c. of dilute nitric acid were then added, and the mixture warmed on the water-bath, and shaken until the oil-layer became bright brown, when the removal of silver phosphide was complete. The oil was separated by filtration through a wet filter and washed; the filtrate boiled for ten minutes, cooled and precipitated with molybdic solution, the phosphorus being ultimately weighed as $\text{Mg}_2\text{P}_2\text{O}_7$. When a small quantity of phosphorus, e.g., 7 mgrs., was used the result was low (78 per cent. of the theoretical). Better results were obtained when working on 15-35.6 mgrs., viz. 93 to 100 per cent.

The process having proved reasonably exact, considering the nature of the case, can be used for the determination of phosphorus in sundry confections used medicinally. The preparations (e.g., chocolate bonbons) are rubbed down with CS_2 in a porcelain mortar, filtered and re-treated until the filtrate gives only a slight brown coloration when shaken with silver nitrate. Oxidation must be hindered as far as

possible by covering the funnel with a clock glass. Silver nitrate solution is shaken with the extract, dilute nitric acid then added, and the CS_2 distilled off. The remainder of the process is carried out as described above. B. B.

The Solubility of Difficultly Soluble Salts. A. F. Holleman. (*Zeits. physik. Chem.*, 1893, xii. 125; through *Chem. Zeit.*)—The author has determined the solubility of salts generally termed insoluble by estimating the electrical conductivity of their saturated solutions, which should, according to the dissociation theory of solution of Van't Hoff, contain free ions only, to the number of which the electrical conductivity is proportional. The following are the results obtained.

Salt.	Parts of water for solution of one part of salt.	Temperature. °C.
BaSO_4	429,700	18.9
"	320,000	37.7
SrSO_4	10,070	16.1
"	10,030	26.1
AgCl	715,800	13.8
"	384,100	26.5
AgBr	1,971,650	20.2
"	775,400	38.4
AgI	1,074,040	28.4
"	420,260	40.4
CaC_2O_4 (? 2 H_2O)	148,220	13.6
"	124,400	24.6
BaCO_3	64,070	8.8
"	45,566	24.2
SrCO_3	121,760	8.8
"	91,468	24.3
CaCO_3	99,500	8.7
"	80,040	23.8

B. B.

The Use of Definite Voltages for Electrolytic Separations. H. Frendenberg. (*Zeits. physik. Chem.*, 1893, xii. 97; through *Chem. Zeit.*)—The author has extended recent work dealing with the electrolytic separation of metals by the use of currents of definite pressures. He classifies metals into three groups: (1) Those which are not separable from their aqueous solutions by currents of moderate voltage, e.g., the metals of the alkalis and alkaline earths—aluminium and chromium; (2) Those which can be precipitated on the anode as peroxides, viz., lead manganese and thallium; (3) Those appearing in the metallic state on the cathode. The voltage of the current has but little influence on the separation of these three groups, but applies chiefly to the separation of members of the third group. The author uses a Gölcher thermo-electric battery because the Daniell, Leclanché, and Meidinger cells give currents of low voltage, and that of the Bunsen is too variable for the purpose in hand, which necessitates an approximately constant electrical pressure. As the voltage, and not the total electrical energy, is to be measured, the use of the oxyhydrogen voltameter, is abandoned in favour of a suitable galvanometer. The separation of various metals was effected, precipitation being conducted in platinum dishes. Thus silver was separated from copper in a mixed solution of

their nitrates—acid with nitric acid—by a current having a pressure of 1·8 volt. Silver was also separated from bismuth from a solution of the nitrates—acid with nitric acid—by a current of the same voltage. Silver was separated from arsenic (1·7 to 1·8 volt), and from antimony (1·2 to 1·3 volt), and from cadmium (1·6 volt). Similar separations were effected for silver from platinum and zinc; mercury from copper, bismuth, arsenic, antimony, and cadmium; and for copper from many other metals. In most solutions the voltage requisite to precipitate copper is 1·8 volt, but in an oxalic solution 1·1 volt suffices. Copper was separated from cadmium and arsenic, and in the case of the latter metal, both in sulphuric acid solution at 1·85 volt, and in nitric acid solution at 1·9 volt.

B. B.

Recovered Rubber. R. Henriques. (*Chem. Zeit.*, 1893, xvii. 1266.)—The importance of the American trade in recovered rubber may be gathered from the fact that about twenty-five million pounds of old overshoes are collected and worked up annually. Two processes of recovery are in use. According to the first, the rubber is finely ground, sifted to separate it from fragments of textile material, steamed under a pressure of six atmospheres, and rolled into plates. In the second method the raw material is divided into pieces about 1 sq. cm. in size by passage between channelled rollers, boiled with dilute sulphuric acid to remove textile materials, washed with water alkaline with soda, finely ground and steamed in the same manner as in the former case. Apparently the steaming process partly devulcanizes the rubber, as otherwise it could scarcely be moulded. This view is upheld by the observation that recovered rubber only contains a little vulcanizing sulphur, but a good deal of sulphuric acid (presumably as sulphates).

Analyses are given of three samples of rubber put upon the market by the Rubber Reclaiming Company, New York, in the form of black rolled plates 3·5 mm. in thickness, possessed of no great elasticity, and, in fact, resembling ordinary black rubber goods of common quality. The material is loaded with mineral matter, as is common for goods of this class, the following figures being obtained :

	A.	B.	C.
Sp.Gr.	1·66	1·59	1·65
PbO	12·87	14·02	12·23
CaSO ₄	22·13	21·59	21·43
CaCO ₃	18·00	10·91	17·86
Fe ₂ O ₃ + Al ₂ O ₃	0·80	2·74	1·10
SiO ₂	1·75	0·40	1·60
H ₂ O	0·52	0·55	0·62
Vulcanizing S	0·71	2·03	1·40

Fatty oils and rubber surrogates were looked for by the author's new methods (*ANALYST*, xviii., 223), but were absent. Asphalt and lampblack were recognised qualitatively, but no exact process for their estimation yet exists.

But little information has been published as to the composition of rubber used for such purposes as the manufacture of overshoes. One recipe which has appeared gives 18 parts of Para rubber, 11 of litharge, 40 of chalk, 3 of asphalt, $\frac{1}{2}$ of lampblack, and $11\frac{1}{2}$ of sulphur. The product would be brittle, however, on account of the small percentage of rubber and the large proportion of sulphur. In other

respects it will be seen to be similar to the mixtures sold as recovered rubber, the composition of which is given above. In this recovered rubber it is a matter of much difficulty to determine with precision the content of true rubber, but it may be taken as in no case higher than 35 to 40 per cent. B. B.

New Volumetric Methods for the Valuation of Fowler's Solution and of Tartar Emetic. S. Györy. (*Zeit. Anal. Chem.*, 1893, xxxii. 415; through *Chem. Zeit.*)—Five c.c. of Fowler's solution (*Liq. Arsenicalis*) are diluted with 10 c.c. of water; 0.5 to 1 gramme of potassium bromide is added, and the whole acidified with 10 per cent. hydrochloric acid; a drop of methyl-orange solution (1 : 1000) is added, and the solution titrated with $\frac{N}{10}$ potassium bromate solution until the red colour disappears. The addition of a drop too much of the potassium bromate solution will be revealed by the yellow colour of the bromine. For the similar treatment of tartar emetic 0.3 gramme is taken, and 25 c.c. of 10 per cent. hydrochloric acid are added. A simplification of the process may be effected by premitting the addition of potassium bromide; for the oxidation of arsenious or antimonious oxide by potassium bromate in presence of hydrochloric acid must yield hydrobromic acid, which will be ready to react with evolution of bromine when an excess of potassium bromate has been added. A. G. B.

Separation of Arsenic from Antimony. L. Garnier. (*Jour. Pharm. Chim.*, 1893, [5], xxviii. 97; through *Chem. Zeit.*)—Dragendorf's method for separating these two metals in toxicological analysis consists in treating the mixed sulphides with ammonia, fusing the undissolved antimony sulphide, still containing some arsenious sulphide, with sodium nitrate, and dissolving any sodium arsenate which may have been formed with water, in which the sodium antimoniate is practically insoluble. The author finds that ammonia always dissolves some antimony sulphide, together with the arsenious sulphide; he therefore recommends Ritter's method. This is performed by dissolving the mirror of arsenic and antimony obtained by Marsh's process in aqua regia and treating the solution with a mixture of tartaric acid, magnesium sulphate, ammonium chloride, and excess of ammonia. The arsenic is precipitated as ammonium magnesium arsenate, and the antimony is thrown down in the filtrate by hydrogen sulphide. A. G. B.

EXTRACT FROM THE REPORT OF THE COMMISSIONERS OF THE INLAND REVENUE.

The following is taken from the report of the Principal of the Somerset House Laboratory :

The systematic examination into the composition of genuine milk alluded to in my last report has been continued during the year, and the results show that while there has been a sensible improvement as compared with the results obtained in previous investigations in the quality of milk as regards the proportion of fat, the percentages of non-fatty solids in the several milks were relatively in agreement with the figures formerly obtained; whatever difference exists is in the direction of a somewhat larger proportion of the milks giving non-fatty solids in amount under 8.6 per cent. It is

now, therefore, more than ever apparent that a limit of 85 per cent. for determining the presence of added water, when judging from the non-fatty solids only, is as high as can be fairly justified, not only in respect to milk from single cows, but also in the case of verified mixed dairy samples.

The results afforded by this investigation, which embrace the milk of 273 individual cows, and the mixed milk of 55 dairies, have been forwarded to the Local Government Board for publication in their annual report.

REFERENCES UNDER THE "SALE OF FOOD AND DRUGS" ACTS.

Fifty-one samples have been referred to us by the magistrates under these Acts. They comprised milk, butter, lard, coffee, whisky, laudanum, pepper, and mineral waters.

Thirty-seven of the samples, or three-fourths of the whole number, consisted of milk. The charge in 24 of the samples was for dilution with water; in eight for abstraction of fat; and in five both offences were alleged to have been committed. Of the samples alleged to contain added water, our results enabled us to support the charge in 22 out of the 24 cases; and of those, where fat was alleged to have been abstracted, we were able to report in a similar way in seven out of the eight cases. Of the five samples where both offences had been charged, we agreed fully with the Public Analyst in four of the cases, but only as regarded added water in the fifth. We could not concur in this case as to the second charge of abstraction of fat, as the sample had been purchased and was labelled as "skim milk," and as it contained much more fat than "skim milk" usually does, we reported that, while the sample undoubtedly contained added water, it could only be considered as deficient in fat when compared with whole milk.

Of five samples of butter, two were alleged to contain foreign fat, and one excess of water; as to the other two we are not aware what offence was charged. The last-named two samples corresponded in composition with genuine butter, as also did one of the samples alleged to contain foreign fat. In the other sample alleged to contain foreign fat, as well as in that where excess of water was charged, we concurred in the conclusions of the Public Analyst.

In two samples of lard, one of whisky, one of laudanum, and one of pepper, we agreed with the public analyst.

In one sample of coffee alleged to contain 94 per cent. of chicory, it was contended on the part of the defendant, who had had the coffee examined by another analyst, that the percentage of chicory was very largely overstated, and this we found to be the case.

In three samples of mineral water, alleged to contain lead, we found that while lead was present, the quantity was extremely minute, and that the metallic contamination was principally tin.

CORRESPONDENCE.

To the Editors of the ANALYST.

GENTLEMEN,—In reference to the criticisms by Messrs. Richmond and Boseley in your last number, on our method of titrating mixtures of milk-sugar and cane-sugar, kindly allow us a few words of explanation.

We do not object to criticism of the method; what we do object to is the failure of these two gentlemen to see that the fluid recommended by us (*ANALYST*, x. 62) contained caustic soda and Rochelle salt, while the fluid they still contend as invalidating our results contained neither of these (*ANALYST*, vi. 218).

We are now content to leave the method to those who care to try it.—Yours faithfully,
ALF. W. STOKES,
R. BODMER.

THE ANALYST.

NOVEMBER, 1893.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on October 4th, at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hahner occupied the chair.

The minutes of the last meeting were read and confirmed.

Mr. George Rudd Thompson, F.I.C., 57, Dock Street, Newport, Mon., was proposed as a Member.

The following gentlemen were duly elected: As Member, Mr. Wallis Jenkins, Hull. As Associates, Mr. John B. Ashworth, Liverpool, and Mr. Benjamin Watmough, Leeds.

The following paper was then read by the author:

DISTINCTION BETWEEN IRON PYRITES AND OXIDE OF IRON IN THE COMMERCIAL ANALYSIS OF MINERAL PHOSPHATES.

By H. H. B. SHEPHERD.

WHEN, in 1891, the Glaser method for the determination of oxide of iron and alumina in phosphates was under consideration by a conference of chemists assembled at the instance of the Chemical Manure Manufacturers' Association, the question as to the acid to be employed as solvent came under discussion, general exception being taken to the use of nitrohydrochloric acid, as directed by Glaser, on the ground that iron, existing in the form of pyrites, would thereby be improperly included in the determination.

Considerable differences between the results of English and continental chemists have lately come to my knowledge which are clearly traceable to this cause, English chemists determining, as a rule, the iron and alumina soluble in hydrochloric acid, and German, French, and Belgian chemists that soluble in a mixture of nitric and hydrochloric acids.

The view taken by most English chemists is very clearly expressed in the following extract from a paper by Dr. Bernard Dyer (*Chem. News*, liii. 51):

"It may have been noticed at the outset I mentioned hydrochloric acid as a solvent, and this suggests a point that may be productive, and often is productive, of analytical discrepancies. Certain phosphates, as is well known, contain iron pyrites. It is not fair to regard the iron in pyrites as oxide of iron; and hydrochloric acid has the advantage of not dissolving up the pyrites, but leaving it amongst the silicious

matter. The solution contains only the iron existing as oxide (or phosphate). The iron present as pyrites may be determined by igniting the sand and treating it with hydrochloric acid, in which the iron is then of course readily soluble, and may be easily determined.

"But no analysis of a phosphate should include in the term 'oxide of iron,' the iron that is present as pyrites.

"If nitric acid be added to the original solvent, the *whole* of the iron passes into solution, and is all included as 'oxide,' unless, of course, a separate determination of pyrites be made, and a deduction made for it.

"If the pyrites is not shown separately in the analysis of a phosphate which contains it (such as the Carolina river phosphates), the statement ought to be, to avoid misunderstanding, either—

"Oxide of iron (exclusive of iron as pyrites), x.x per cent.,

"or—

"Oxide of iron (inclusive of iron existing as pyrites), x.x per cent.

"But it is far better to state the pyrites and oxide of iron separately, that there may be no confusion. The point is important not merely for avoiding discrepancies, but as materially affecting the apparent value of a phosphate; for iron pyrites in a phosphate is by no means the objectionable constituent that oxide of iron is."

I must acknowledge my indebtedness to my friend for this somewhat free use of his paper. It possesses, however, this special interest, viz., that it was written as a contribution to the discussion of the subject upwards of seven years ago, and, as an expression of opinion on the pyrites question, has remained unchallenged by English chemists, so far, at any rate, as published papers are concerned, ever since.

I may mention, however, in passing, that Mr. W. W. Mallon proposed at about the same time (*Chem. News*, liii. 85) the adoption of dilute sulphuric acid as the solvent, in order to imitate as nearly as possible the actual superphosphate manufacture; but this, though possibly of interest from the point of view of the action of the solvent upon the clay usually present, does not, if considered as an alternative to

the use of nitric acid, affect the point at issue. There can be no doubt that amongst most English chemists a decided opinion has been formed against the inclusion of the iron of pyrites in these determinations, based upon its insolubility in the process of superphosphate manufacture, but it does not appear that the distinction between this form of iron and iron in other combinations has hitherto been considered to the same extent on the continent. In none of the published processes can I find any special reference to the question of pyrites, and I can only infer, from the general directions given for the use of nitric acid, that its inclusion is intended.

I have been informed, however, that an attempt at justification has lately been made by a German chemist in a communication to his clients, on the grounds that oxides of nitrogen are present in small quantities in the chamber acid used by superphosphate manufacturers, and though I confess to have been biased against this as a serious argument, I have, nevertheless, endeavoured to give it impartial consideration. Now, Lunge ("Sulphuric Acid and Alkali," p. 466) gives a table on the authority of Kolb, showing the amounts of nitrous and nitric acids present in chamber acid under different circumstances, from which I gather that under normal conditions the sum total of both nitrous and nitric acids does not amount to more than about 0.03 per cent.

I have myself examined a good many samples of chamber acid, and am convinced that under normal working conditions the total amount of nitrous and nitric acids cannot be regarded as more than a trace. Frequently it is only sufficient to give the faintest possible reaction with ferrous sulphate. The following are some results which I have recently obtained on testing the acid from the various parts of a set of chambers with towers in good working order :

				Total nitrosity in terms of N_2O_5 .
No. 1 chamber	0.002
No. 2 "	too small to determine
No. 3 "	0.002
No. 4 "	0.001
Denitrated acid from the Glover tower	0.003

The amount, of course, fluctuates within certain limits, but I maintain that the average nitrosity is quite insufficient to act upon the pyrites in the way alleged.

The following tests were recently made in samples of acid used for dissolving, obtained from different Works :

				Total nitrosity in terms of N_2O_5 .
No. 1 Works	0.022
No. 2 "	0.004
No. 3 "	0.003

I have, however, carried the inquiry a step further by submitting the matter to an actual practical test. At the works with which I am connected we lately received a parcel of South Carolina river phosphate of the following analysis :

Phosphoric acid	27.37
Iron in form of pyrites	0.77

With this material I made an experimental superphosphate mixing, on a manufacturing scale, using equal parts of phosphate and chamber acid of 110° Tw.

The superphosphate showed on analysis the following results :

Total phosphoric acid	14.49
Iron in form of pyrites	0.42

Now, in order to compare the pyrites iron found unaltered in the superphosphate with that originally present in the raw phosphate it is necessary to make a correction for the decrease in weight due to loss of carbonic acid, water, etc., resulting from the mixing.

Of the two ways of determining this loss, viz., by weighing the resulting superphosphate, or by calculation from the total phosphoric acid found in it, I prefer the latter as being more exact, and this gives a loss in the present instance of 5.60 per cent.

The comparison, after making the necessary correction, will therefore stand as follows :

Pyrites iron in the materials before mixing	...	0.41
" " after "	...	0.42

The plea advanced in Germany appears, therefore, to be groundless.

It is only fair, however, to state that I have succeeded in dissolving a small proportion of the iron of pyrites in a phosphate by chamber acid, but only under conditions that could never possibly occur in practice. By long digestion in a very large excess of an acid of unusually high nitrosity, and at a higher temperature than occurs in the actual manufacture, I actually effected the solution of 0.19 per cent. of pyrites iron.

Many chemists may not be aware how seriously the trade is affected by differences due to the divergence in practice referred to.

It is the custom for sellers to guarantee a maximum percentage of oxide of iron and alumina, with the proviso that, should the guarantee be exceeded, an allowance shall be made of two units of phosphate of lime for every such unit of oxide of iron and alumina in excess of the guarantee. In some cases, however, and notably when French or American phosphates are dealt in, the guarantee goes even beyond this. A maximum of 3 per cent. is guaranteed with compensation as above up to 4 per cent., but should this limit be exceeded buyers have the option of rejecting the entire cargo. It is easy to see what important issues may therefore depend upon the question of the exclusion or inclusion of the iron of pyrites in the determination. A phosphate containing but 3 per cent. of oxide of iron and alumina may by the inclusion of the iron of the pyrites be brought over the 4 per cent. limit, with the result of the rejection of a cargo involving consequences amounting possibly to thousands of pounds.

We have to bear in mind in these questions that the chemist's certificate is required, principally, if not solely, as a basis of price between buyer and seller, and that the want of a uniform system renders one party liable to gain an unfair advantage at the expense of the other.

As it is so clearly desirable that we should endeavour to come to an understanding with our continental friends upon the subject, I should be very glad to receive any assistance in the way of suggestion or otherwise that might help towards the attainment of this end.

DISCUSSION.

Mr. Otto Hehner said that, as there were several members of the Society present who had a wide experience in the matter brought forward by Mr. Shepherd, he hoped they would take part in the discussion. As to the international arrangement which had been suggested, he should be pleased to see it brought about; but, in any case, the publication of the matter would doubtless tend to remove the discrepancies which existed at the present time.

Mr. Hermann Voss said that as a manufacturer he would not discuss the more scientific part of the matter, but perhaps he might be permitted to say a few words about the commercial side of the question. As had been stated in the paper, it was a very important question, not only for the merchant, the buyer, and the seller, but also for the manufacturer in the end, because on the correctness of the determination of iron and alumina depended the calculation as to the mode of manufacture, and also the profit or loss on the manufactured article, because the manufacturer had to

guarantee the presence of so much soluble phosphate in the superphosphate, and if the first determination of iron and alumina in the phosphate was not quite correct, or was misleading, he would not be correct in his calculation as to what was the result he might expect in the manufactured article, and it might ultimately turn out that he would have to pay for deficiency. He was in a position to state that recently the differences among results alluded to in the paper had been very great. At a meeting of chemists and the trade an endeavour had been made to find out a way by which more uniform results could be obtained. He thought that the meeting of chemists held in London had been productive of good results, inasmuch as it brought the results of the English chemists nearer than they were before. But since the introduction of the Glaser method, and the various modifications thereof, it seemed to have had the effect of again bringing out differences not only between the results of continental chemists, but also between the results of English chemists, and he believed the tables which he had had prepared would show that the differences in the results in the iron and alumina determinations in commercial phosphates were very great.

OXIDE OF IRON AND ALUMINA TESTS IN RECENT SHIPMENTS OF SOMME PHOSPHATES.

Cargo.	Chemist.	Oxide of iron and alumina combined. Total.	Oxide of iron.	Alumina.
No. 1	English	2.20	1.23	0.25
	French	1.48		
No. 2	English	3.60	1.35	0.63
	French	1.98		
	German	3.24		
No. 3	German	2.64	1.23	0.21
	"	3.85		
	French	1.44		
	English	2.90		
No. 4	French	1.54	1.29	0.25
	English	2.62	1.10	1.52
	"	2.70		
No. 5	German	3.91	1.60	0.89
	"	3.67		
	Belgian	2.33		
	French	2.49		
No. 6	French	2.28	1.36	0.92
	English	3.15		
	"	3.17		
No. 7	English	3.95	1.76	2.13
	"	3.89		
	French	2.93	1.77	1.16

OXIDE OF IRON AND ALUMINA TESTS IN RECENT SHIPMENTS OF OSSO PHOSPHATES.

Cargo.	Chemist.	Oxide of iron and alumina combined. Total.	Oxide of iron.	Alumina.
No. 1	French	3.98	2.36	1.62
	Belgian	3.72		
No. 2	English	5.96	1.79	1.63
	Belgian	4.74		
	French	3.42		
No. 3	English	4.97	3.06	1.84
	"	5.40		
	Belgian	3.75		
No. 4	French	4.90	2.50	3.43
	English	5.50		
	"	6.10		
No. 5	Belgian	3.40	2.86	3.08
	English	5.93		
	"	5.94		
No. 6	Belgian	4.97	2.02	0.71
	"	4.20		
	English	4.76		
No. 7	Belgian	3.23	2.02	0.71
	French	2.73		
No. 8	English	4.03	2.02	0.71
	French	2.73		

OXIDE OF IRON AND ALUMINA TESTS IN RECENT SHIPMENTS OF RIVER PHOSPHATE.

Cargo.	Chemist.	Oxide of iron and alumina combined. Total.	Oxide of iron.	Alumina.	Iron pyrites determined separately.
No. 1	German	2.30	1.14	0.67	0.52
	"	2.65			0.23
No. 2	English	1.81	1.14	0.67	2.46
	German	3.27			
No. 3	German	2.95	1.50	1.45	0.26
	"	2.84	2.14	0.70	0.39
No. 4	English	2.02	0.60	1.42	0.52
No. 5	English	2.16	1.45	0.71	1.57
	"	2.32	1.70	0.62	
	"	3.00			
No. 6	English	2.19	1.31	0.88	0.84
No. 7	English	1.62	1.26	0.36	2.63
	"	1.43			
No. 8	English	2.32	1.11	1.21	1.11
	"	2.30			
No. 9	English	1.60	1.24	0.36	2.61

At that meeting it was said that the results should not vary more than a half per cent., and it would be quite enough if in a contract for phosphates it was stated that if a difference of a half per cent. existed the matter should be referred to a third chemist. The figures in the tables sometimes amounted to as much as 2 per cent., which could not be due to variation in the sample. It could only be traced to the different methods employed. He thought it would be self-evident that the trade, the manufacturer, as well as the merchant, were very often at a loss to know where the truth really lay. He did not know who was right and who was wrong, but he thought that it was in the interests of science, especially of the science of chemistry, that such serious differences should disappear. The greatest difference occurred in phosphates sold in either France or Belgium, because they were sold on the mean of the results obtained by French or Belgian chemists, and on those obtained by English chemists, the differences sometimes amounting to over 2 per cent. As the author of the paper had said, there was a guarantee of 3 per cent., with a maximum of 4 per cent., and if this maximum were exceeded it happened that cargoes were thrown on the hands of the seller, the sums involved sometimes amounting to thousands of pounds. As an illustration, he would refer to one case where the average percentage of iron and alumina amounted to 4.05. The maximum guarantee was exceeded, and consequently the buyer had a right to throw up the whole cargo, which consisted of 2,500 tons. The price of the phosphate had dropped 12s. to 15s. a ton, and there was a further difference claimed of 5s. or 6s. a ton. Finally the matter was settled by an allowance of £2,500—for the sake of 0.05 per cent. This sort of thing only happened on rare occasions, and he was quite sure that not many buyers would be found who would, for the sake of 0.05, claim a difference of £2,500. With regard to iron pyrites in phosphates, he believed, as is shown in the paper, that manufacturers need not fear the presence of such pyrites, and therefore it should not be quoted amongst the noxious ingredients of the phosphate. It would be found that in one of these tables there was an analysis by an English chemist who obtained 1.81 of iron and alumina, whilst a German chemist found above 3 per cent. in another sample of the same cargo. The disparity could not be explained by the difference in material, or by errors in analysis; it could only be explained by the custom of employing different methods of analysis. Although attempts had been made to get continental chemists to adopt the same method as that employed by English chemists, the former seemed to think that their method was a better and a more correct one; and as merchants here bought phosphates on the continent, they had to accept the figures. He was quite convinced that if chemists in this country would set the ball rolling, the matter would be seriously discussed on the continent, and the best method of analysis would then be arrived at.

Mr. John Hughes confirmed what had been said by Mr. Shepherd. From the remarks which had fallen from Mr. Voss he gathered that it must be a practice, at least among certain chemists on the continent, to determine and include the iron of the pyrites in the total oxide of iron, without making any distinction, and if that was the case, the members of the Society should take the opportunity of stating very plainly that, among English chemists, it was not the practice to recognise the presence of pyrites as in any way injurious in the manufacture of superphosphate.

He himself had never included the iron of the pyrites when reporting his determinations of iron. He had had in the past a great deal of experience in the matter of the treatment of river phosphates containing pyrites during the time he was associated with Messrs. Lawes and Co., and he had ample opportunity of testing whether or not the pyrites was affected by the action of the acid. He found that no appreciable action took place during the mixing, as mentioned by Mr. Shepherd; but if chamber acid were taken, and the sample was treated with an excessive quantity of this acid, in some cases it was possible to oxidize a considerable portion, if not the whole of the iron of the pyrites; but in the manufacture of superphosphate that was not the case, the conditions being different, and the phosphate, and not the acid, being in excess. Regarding what Mr. Voss had said in reference to the matter of variation in the results obtained by English, as compared with those obtained by continental chemists, he would like to mention that at a meeting of manure manufacturers some years ago he took up the same line of suggestion, namely, that when gentlemen of business reduce to writing certain prices for the sale of certain products they should themselves take a little trouble, first of all, to find out what the limits really ought to be as regards this objectionable iron. He had frequently been called in to assist in disputed cases; but had he been called in to advise, before the contract was made, no difficulty would have arisen. As regarded the case mentioned by Mr. Voss, had the sellers of the phosphates taken the trouble to have the cargo carefully tested, not once, but several times, they would have been able themselves to know how far they could go in guaranteeing the quality of the phosphates as regarded the presence of iron and alumina. He therefore suggested that whilst chemists should, of course, endeavour to be as accurate as possible in their determinations, it was only fair that the manufacturers themselves should take a little trouble, and give the chemist an opportunity of making an analysis and of saying whether or not they could guarantee the materials to be of a certain quality.

Dr. Bernard Dyer also confirmed what had been said by the author of the paper. He was of opinion that the confusion between oxide of iron and iron pyrites ought long before now to have been done away with. On the continent it was unfortunately the practice to work more by stereotyped methods. For example, a process for the determination of oxide of iron in phosphates was proposed at a convention of agricultural chemists, and it probably did not occur to anyone present that some phosphates contained pyrites. The process began with the adoption of nitric acid as a solvent, simply because it was a useful solvent, for quite another purpose, viz., for determination of phosphoric acid by molybdenum. The process was then adopted formally and officially, and every chemist in Germany worked by it, and when he came across a phosphate containing pyrites he was bound hand and foot. He had to follow out the prescribed process; he used nitric acid as a solvent, and obtained a result which was altogether wrong. The fact that pyrites was not acted upon to any appreciable extent in superphosphate making, which formed the backbone of Mr. Shepherd's paper, was brought home every day to analysts who had to deal with superphosphates made from phosphates containing pyrites, for they constantly found in such superphosphates pyrites in an unchanged state. Mr. Shepherd's figures showed that practically the whole of the pyrites was left unacted

upon by chamber acid. The suggestion made by Mr. Hughes to the effect that more explicit statements should be made in contracts would be a very desirable one to follow. It surely ought not to be difficult to express in a contract that the oxide of iron and alumina clause should not refer to iron that existed in the form of pyrites. This plain stipulation should be put into the contract, and the chemist who had to carry out the analysis, whether here or abroad, should have that placed before him in writing at the time he received the sample. Confusion would then not occur. He himself had been of opinion all along, and he had expressed it frequently, that the main differences that occurred between different determinations of oxide of iron and alumina were due, mostly, to the employment of different solvents. He thought that when the iron and alumina had got into solution the differences which might legitimately occur in determining their quantities need be very small. In some phosphates a great deal of clay or kindred aluminous mineral was present, and if merely dilute hydrochloric acid were used the clay was scarcely touched, although the mineral was readily attacked by chamber acid. Evaporation to dryness, with a proper quantity of strong hydrochloric acid, produced practically the same result as the chamber acid. He hoped that Mr. Shepherd's paper, and the discussion on it, would find its way into the analytical journals all over the continent, and that discussion would thereby be aroused abroad as well as at home.

Mr. Shepherd, in reply, said he was pleased to find there had been no adverse criticism on his paper, and thanked Dr. Dyer and Mr. Hughes for their emphatic expressions of opinion in support of the English system of analysis. He thought that Dr. Dyer's surmise as to the origin of the use of nitric acid in Germany was very probably correct, viz., that it was taken in the first instance simply on account of its general use as a solvent for analytical purposes, and that the practice having been begun, was continued, quite irrespective of the action of the acid upon the pyrites, which seemed, indeed, until attention was recently directed to it, to have been altogether overlooked. In face, however, of the present attitude of continental chemists on the matter, he did not think that Mr. Hughes' suggestion to insert a clause in contracts directed against returning pyrites iron as oxide of iron could be carried out. Merchants themselves, moreover, had not always the requisite knowledge at their command to enable them to form a correct opinion upon a purely chemical question, and their interests as buyers and sellers were conflicting. It was only natural, therefore, that they should appeal to chemists for that guidance which it seemed to him particularly within their province to give. No doubt if English chemists expressed themselves freely and forcibly upon the subject, their views would receive attention on the continent. English chemists, however, it seemed to him, stood somewhat at a disadvantage in comparison with their continental brethren, inasmuch as their views could only be expressed individually, and he thought the time was approaching when the continental plan of annual conventions would have to be tried in this country. He suggested that if the Society of Public Analysts or any other recognised body of chemists would take steps in that direction they would earn the gratitude of the mercantile community.

Mr. Richmond then read the following paper :

THE DISCRIMINATION BETWEEN ABNORMAL AND ADULTERATED MILKS.

By H. DROOP RICHMOND.

THE long-continued drought and consequent scarcity of fodder during the summer of 1893 have not been without influence on the composition of milk, and cases have occurred in which the figures given on analysis by genuine milks have fallen below the limits recommended by the Society of Public Analysts to a notable extent. As some of these cases have been used for defending adulteration cases, public analysts may be influenced to adopt a larger margin of safety in interpreting their analytical results, and as a consequence allow larger amounts of adulteration to pass unnoticed. I have collated the results obtained during the present summer, in order to see whether any difference exists between abnormal and adulterated samples.

Though my samples have been neither very abnormal nor as strictly authenticated as some of the samples which have been in the hands of other analysts, I am induced to bring my results before the Society, on account of recent evidence given in court by chemists who are popularly looked upon as authorities on milk-analysis, in which the statement was made that it is impossible to distinguish between abnormal and adulterated samples. As I have had the opportunity of examining comparatively few abnormal samples, I cannot traverse this statement, but I have some evidence that it can be done.

I class as abnormal all samples which fall below the limits of the Society, and divide them into two classes : (1) Those deficient in fat, and (2) those deficient in solids-not-fat.

Class 1. If an analyst finds a sample low in fat, it may be due to three causes :

- (1) Abnormal milk.
- (2) Wilful adulteration.
- (3) Separation of cream during delivery.

It has been decided by the Court of Appeal that a milk-seller is bound to deliver milk as it comes from the cow, and therefore, by this decision, the third cause comes under the Sale of Food and Drugs Act. A suggestion that this might have occurred is manifestly not a good defence, though doubtless if the defendant adduced evidence that this had actually occurred through no gross negligence on his part, no magistrate would impose the maximum penalty ; a defence of this kind would throw no discredit on the public analyst, and after the Lord Chief Justice's ruling he would not be justified in making allowance for this. The defence recently made, that a part of the cream is churned into butter during delivery, is similarly rendered invalid ; indeed, I have found in some thousands of experiments that no appreciable amount of fat is thus removed from the milk by this cause, even in the hottest weather.

It is only necessary to distinguish between adulteration and abnormality. There exists at present, to our knowledge, no method of conclusively deciding between these two ; it is a general, but not universal, rule that low fat in genuine milks is accompanied by low solids-not-fat, and as, moreover, the removal of fat increases the solids-

not-fat, a low fat with high solids-not-fat would give considerable evidence of removal of cream. Any specific gravity of 1.033 or upwards with a low fat is suspicious. As the cream removed contains the larger fat globules, careful microscopical examination will also assist, but the micrography of milk is too imperfectly known to admit of great weight being attached to this evidence. It is perhaps premature to speak of chemical evidence, though the solids-not-fat of separated milk differ to a small extent from those of whole milk. When milk is run through a separator, a thick slime is deposited on the sides of the drum, and this by no means consists wholly of dirt; it is possible that by submitting suspected samples to centrifugal action this slime could be separated and estimated, and from a deficiency of this the presence of separated milk might be presumed.

I may add that the past season has not been characterized by an abnormal lowness in fat, and at the present time I am unable to find any samples with less than 3 per cent. of fat.

Class 2. The known constituents of the solids-not-fat are milk, sugar, proteids (of which casein and albumin are the best known, though others have been described), and salts. The proportion of these is given by Vieth as sugar : proteids : ash = 13 : 9 : 2. My experience shows that this estimate is a very near approach to accuracy.

The Society of Public Analysts in their limit take no account of the separate constituents of the solids-not-fat, but group them together as a whole. Samples do occur which contain less than 8.5 per cent. of solids-not-fat, and these have been specially noticed this summer. Doubtless the cry will be now raised that the time has come to modify these limits. To lower the limit to such a figure as would, while covering the very rare cases of abnormal milk, allow a considerable average adulteration, and would be an act of cowardly betrayal of the honest milk-dealers, who, I venture to think, do not form a minority. It would also play into the hands of unscrupulous tradesmen, and stultify the profession of analytical chemistry.

I have endeavoured to see if the abnormality of these low milks extends further than a deficiency of solids-not-fat. Before the days of Wanklyn, it was the custom to examine the milk more fully than is now done, and to estimate sugar, proteids (collectively or individually), and ash. In my experience the most constant figure in normal milks is the proportion of the ash to the solids-not-fat, which averages 8.3 per cent. It very rarely exceeds the limits of 8.0 to 8.5; in cases of low solids-not-fat this proportion has been disturbed, and the ash has borne a higher ratio to the solids-not-fat. In no case has the percentage of ash in the milk fallen below 0.7 per cent., even in milks notably below the limit. In an adulterated milk containing (say) 8.0 per cent. of solids-not-fat, the ash would be usually lower than this—about 0.66 per cent.; this difference is small, but as the ash is capable of being estimated with great accuracy, it is significant. Other observers have found the same thing; thus Lloyd (*Journ. Chem. Soc.*, 328, 201) finds an average of 0.78 per cent. of ash in a series of abnormal milks as low as 7.5 per cent. of solids-not-fat, and Lowe (*ANALYST*, xviii., 6) 0.94 per cent. of ash in a milk containing 6.0 per cent. of solids-not-fat; De Hailes finds a low ash in one sample (*ANALYST*, xviii., 7), but as his analyses

are very far from agreeing with the formula of Hehner and Richmond, we are justified in considering his work unreliable. Of course, any mineral additions to the milk increase the ash, but there would be no serious difficulty in detecting these in a good many cases, still it cannot be done with certainty in all. The ash of milk is free from carbonates and borates, and (practically) sulphates; the soluble ash is about 30 per cent. of the total ash. Were a milk of low solids-not-fat with a normal ash found, and the soluble ash found to be excessive, of marked alkaline reaction, or containing borates or sulphates in notable amount, the sample may safely be condemned.

In a previous paper I have stated that the deficiency in abnormal milks lies chiefly in the milk sugar; this I have found to be generally true in samples examined this year. The proteids do not decrease to a marked degree; it will be useful in cases of doubt to estimate these. I cannot, however, lay very great stress on these determinations, because not only is the inaccuracy somewhat uncertain, but my experience is limited to about a dozen samples. I am inclined to think that a determination of the total nitrogen is useful, any quantity below 0.5 per cent. being very suspicious. I may incidentally point out that the most reliable data in the analysis of decomposed samples are total nitrogen, ash, and, of course, fat, provided always that a fair average sample can be obtained.

There is one other property of milk which may afford considerable evidence of adulteration—the absence of nitrates in genuine milk. From evidence given in police courts, it seems that the farmer is responsible for nearly all the adulteration detected; since, from the analysis of numerous water supplies on farms, I find that considerably over half the farms of England are supplied with water containing appreciable amounts of nitrates, there is a great probability that adulteration may be detected by finding nitrates in the milk. A method of detecting nitrates which I have found successful, and which Boseley and I have previously roughly described, is this: Place a small quantity of diphenylamine at the bottom of a porcelain basin, and add to it about 1 c.c. of pure (about 97 per cent.) sulphuric acid; allow a few drops of the serum, obtained by adding a little acetic acid to the milk and warming, to flow down the sides of the basin and over the surface of the acid. If a blue colour develops in the course of ten minutes, though it may be faint, it shows the presence of nitrates; after ten minutes a reddish-brown colour is always developed from the action of the acid on the serum. I have detected the presence of nitrates in a milk purposely mixed with 5 per cent. of water, containing 5 parts per 100,000 of nitric acid, and in two cases where I have found nitrates, I have been able to prove the addition of water by other means. One sample which was watered did not contain nitrates, and in this case I examined the water supply and found it free from nitrates.

In conclusion, I think it right to state that abnormal samples occurred in June, July, and August of this year; but now the quality of milk in all parts of the country has greatly improved, and is at least as high, if not higher, than last year. In my opinion no justification exists for making any allowance for meteorological vagaries.

DISCUSSION.

Dr. Bernard Dyer remarked that Mr. Richmond did not seem to be aware of the fact that a number of years ago Mr. Hehner had proposed the use of

diphenylamine as a test for nitrates in milk with a view to detecting the addition of water.

Mr. Richmond, replying to Dr. Dyer, said that Mr. Hehner had on several occasions told him that he had not been able to make the test work well; and, therefore, he (Mr. Richmond) had thought it desirable to fully describe the mode of manipulation which had given good results.

Mr. R. Bodmer said that Mr. Richmond having stated that he considered it was the farmer who adulterated milk, and not the milkman, he must have a very high opinion of the retail London milkman. He was not speaking of large wholesale companies, but of little petty milkmen, who supplied poor districts, such as Bermondsey. Surely Mr. Richmond did not think that such a man was above putting water in milk, and, supposing he did, he would use London water, which contained such a small quantity of nitrates that the nitrate test would be quite useless. He wished to know what process the author of the paper adopted for determining milk sugar; and also how the ash was determined; whether he used a muffle or an argand burner. In one case he (Mr. Bodmer) was told that an analyst ignited his milks in a muffle at almost a bright-red heat, and naturally got only half the normal amount of ash.

Mr. Cassal, in view of the fact that most London waters contained, as a rule, about 0.18 parts per hundred thousand of nitrogen as nitrates, desired to ask Mr. Richmond whether, supposing that a milk had been adulterated to the extent of 10 per cent. with London water, containing that amount of nitrates, he was prepared to say that the test suggested by him would detect that amount. He observed, in reference to Mr. Richmond's remarks concerning the microscopic examination of milk, that, at one time, all the samples of milk which came into his laboratory were examined microscopically; and he might state that, in cases of extensive deficiency in fat, it was invariably found that the fat globules were all very small. In these cases of considerable deficiency in fat, the microscopic examination gave positive evidence of fat abstraction or deficiency by the smallness of their number, and of their size, so long as the examinations were made by the same person, and in the same way; but whether it was possible to found an invariably reliable test on this, he was disposed to doubt. He understood Mr. Richmond to suggest that microscopic examination might enable analysts to say definitely that fat had been extracted from milk owing to the absence of all large globules. He doubted whether a public analyst could go into court with such evidence. With regard to the nitrate test, it struck him (Mr. Cassal) as being a matter of the utmost importance, and very well worth working out, although the suggestion was by no means a new one. Several years ago Mr. Uffelmann suggested testing for nitrates in milk for the purpose of detecting added water. He believed that the proposition was received at the time with some doubt, not to say with some ridicule, a fact which was to be regretted. He did not think that the detection of the presence of nitrates in milk adulterated with London water to the extent of 10 per cent. should at once be put on one side as impossible. In the case which occurred recently in a London police-court, the so-called evidence for the defence could without much trouble have been proved to be

absolutely valueless; but the detection of anything which could be regarded as characteristic of extraneous water, beyond what was now relied upon, would plainly have been of great value in its effect on the magistrate's mind. He was glad that Mr. Richmond had alluded to the evidence given for the defence in that case, and that he had expressed his opinion that it was worthless. He (Mr. Cassal) would invite the Society to carefully consider the point that had been raised in a recent case in which a certain analyst, not a member of the Society, was called to give evidence on behalf of a vendor of milk alleged to be adulterated. This analyst was reported to have said that he had seen seventeen cows milked in a field, that he had analysed a sample of the mixed milk, and that if such a sample had reached him in the ordinary course he would have reported it as containing from 8 to 10 per cent. of added water. It was unfortunate that in the report of that case no statement was made as to whether this analyst was cross-examined with respect to the limit which he used, because it would have been perfectly possible to make an assertion of that description by taking a high standard; and he (Mr. Cassal) was disposed to believe that if that analyst had been so cross-examined he would probably have had to admit that the limit he used was not the same as that employed by the public analyst, who had certified the milk in dispute to be adulterated. But while public analysts would hail with all possible satisfaction any valuable addition to the methods employed for detecting the difference between added water and the water which is natural to milk, the position of the public analyst under the Sale of Food and Drugs Act in regard to milk was quite clearly defined, and was not affected by these so-called abnormal cases. The composition of the article which a purchaser was entitled to get when he asked for milk, was perfectly well-known from the hundreds of thousands of analyses of milk that had been made, and it was the duty of public analysts to point out that such abnormal samples as had been alluded to were not *milk*, either from a scientific, legal or public point of view. It was not the article required by the purchaser, but something totally different. It seems to be accepted in a loose kind of way by many persons that milk was anything which could be extracted from the udder of any cow. Such a definition was utterly unscientific and obviously worthless. The only possible definition was one founded upon the large number of analyses made of the normal article, which showed beyond all question what the composition of that normal article was. The results of analyses of abnormal fluids were certainly highly interesting in themselves—especially from a pathological point of view—but they did not affect the position of the public analyst at all, because such fluids were not milk in the scientific, legal or commercial sense. In the case alluded to by Mr. Richmond, viz., *Dyke v. Gower*, it was laid down by the High Court that milk must be sold whole by the milkman, and that the defence that the fat was deficient because the cream had risen in the can during the process of serving was no defence. It had, therefore, been definitely settled that the vendor of milk was responsible that the article he sold was what was demanded of him, and this dictum was clearly applicable to the case of watered milk, as well as to the case of fat abstraction. It was, in fact, the business of the producer or vendor of milk, or of any other article, to see that he sold the article that he was called upon to sell. In the recent case alluded to it was therefore plain that under the 6th section of the Sale

of Food and Drugs Act, an offence had been committed, and that a conviction should have followed. He could only express the hope that all public analysts would take up this position, which was, in fact, the only scientific and logical position, and would refuse to recognise as milk any of those abnormal fluids and disease-products, the composition of which had been on certain occasions laid before the Society. He desired, further, to express his deep regret that there were still persons to be found who were willing to give a certain type of evidence in Courts of Law.

Mr. Cecil H. Cribb mentioned that in a case in which he had recently been concerned he was asked, in conjunction with another chemist, to see eight cows milked, and to analyze samples of the milk from each afterwards. No less than twelve out of sixteen samples turned out to be abnormal, and it was interesting that in the case of one of the cows both the morning and evening milk gave figures below those obtained by Mr. Richmond. The figures being for the evening milk :

Total solids	11.01
Fat	3.54
Sugar and proteids	7.47
Ash	0.69
							8.16

The results of the rest of the abnormal samples varied between those just quoted and the normal. Without going into details then, though he would like to have the figures recorded in THE ANALYST, he observed that the relation between the ash and the solids-not-fat in all cases bore out Mr. Richmond's suggestion.*

Mr. E. J. Bevan said that he had tried the test mentioned by Mr. Richmond, but in a sample of what he had reason to believe was genuine milk he had certainly got a reaction for nitrates. He would like to ask Mr. Richmond whether nitrates might not often find their way into milk. In the case referred to by him he tested other milks side by side, and in these instances he obtained no reaction whatever, and he could only come to the conclusion, subject to the limited experience he had had in this particular test, that there were nitrates present in the milk which he had examined, and which he regarded as genuine. He admitted that if in testing a milk one got a very considerable reaction for nitrates, it might throw a good deal of suspicion on the sample. He observed that Mr. Richmond had not noticed any recent

* The following are the figures for each of the abnormal samples, the analyses of the morning and evening milk of the same cow being placed together :

		Total Solids.	Fat.	S.N.F.	Ash.	Ash x 100. S.N.F.
(1)	{ Morning	11.01	3.54	7.47	0.70	9.4
	{ Evening	11.16	3.73	7.43	0.70	9.4
(2)	{ Morning	10.97	3.06	7.91	0.73	8.8
	{ Evening	11.77	3.97	7.80	0.76	8.8
(3)	{ Morning	11.12	3.20	7.92	0.69	8.7
	{ Evening	11.54	3.80	7.74	0.69	8.9
(4)	{ Morning	12.16	4.04	8.12	0.70	9.2
	{ Evening	12.10	3.99	8.11	0.70	9.8
(5)	{ Morning	11.63	3.47	8.16	0.72	8.6
	{ Evening	12.33	4.16	8.17	0.72	8.6
(6)	{ Morning	11.89	3.42	8.47	0.69	8.1
	{ Evening	12.61	4.21	8.40	0.71	8.4

The cows were considered to be in their usual health. It was suggested that the change from the winter food (oil-cake, etc.), to fresh grass, which had recently taken place, was the cause of the pooriness of the milk.

diminution in the amount of fat in normal samples. He was exceedingly pleased to hear this, because it entirely bore out the results of his own experience. He had tried to convince Mr. Plowden, one of the London magistrates, that no natural diminution had occurred, and offered to show him the results of analysis of 188 samples taken in July, August, and September. The average percentage of fat in these samples, which included both genuine and adulterated, was 3.6, and in the particular case in which he had to appear the percentage was 2.4. The magistrate refused to listen to any such arguments, or to look at any results, or take any further evidence, and the case was dismissed.

Mr. Hehner thought that until the nitrate test had been used by many analysts, and under a variety of varying circumstances, it would hardly be safe to rely upon it as a definite test for adulteration with water. The subject brought forward by Mr. Richmond was of the utmost importance. The way out of the difficulty suggested by Mr. Cassal—namely, that milk which, though unwatered, was not of the normal minimum composition, and should not only not be called milk, but that the sale of it should render the vendor liable to punishment under the Food Acts—would hardly at present commend itself to magistrates. In time Mr. Cassal might be able to educate the public to look upon milk as the normal fluid produced by healthy, well-fed cows, but as yet the time had not yet ripened for the criminal prosecution of persons selling abnormal, but unadulterated milk. Meanwhile, Mr. Richmond was looking upon the facts as they were, and had made a very gallant attempt to grapple with the question of abnormal milk. He (Mr. Hehner) thought, however, that he had hardly been successful. As to the suggestions just touched, but not insisted, upon by Mr. Richmond, concerning the size of the fat globules and the presence of deposits in centrifugals, he would say that the size of the fat globules did not depend upon the amount of skimming alone, but was materially influenced by the period of lactation in the cow. When a cow was in full milk the globules were comparatively large, but their size gradually diminished, until, when the end of the period of lactation approached, their size was very considerably reduced. It must be noted in connection with this that it was just towards the end of the lactation that cows gave the worst milk and the poorest butter, and that that was just the time when the difficulty of distinguishing between milk naturally poor and that artificially reduced in quality arose. With reference to the deposit in milk-separators, this, as far as he remembered, had first been mentioned by Mr. Faber at a recent meeting of the Society. Considering that Mr. Richmond had not made any determinations of the amount of this deposit, nor even ascertained its exact nature, it appeared to him (Mr. Hehner) extremely risky to found upon such indefinite knowledge such a proposal as Mr. Richmond had done.

As to Mr. Richmond's arguments founded upon various constituents of the solids-not-fat, the case was considerably stronger. The main contention advanced was that the amount of ash in genuine milk, however low in solids-not-fat that milk might be, was fairly constant. Mr. Richmond agreed with Dr. Vieth in this; the latter found that the ash bore a constant, or nearly constant, proportion to the percentage of caseine, while Mr. Richmond comes to the conclusion that in abnormally low milks the caseine is not much affected, but that it is the milk-sugar that diminishes. Or,

putting it in other words, the ash bore, in genuine milk, a fairly constant proportion to the amount of solids-not-fat, being about 8.3 per cent. of the latter, while in abnormally poor milks the percentage of ash to solids-not-fat was higher. Generally speaking, all this might be perfectly true, but it would not help the Public Analyst much in coming to a correct judgment; for it was in the samples containing about 8 to 10 per cent. of water where the difficulty of judging came in, and in these the ash was of very little definite help. Assuming a milk with 8.5 per cent. of solids-not-fat contained 0.73 per cent. of ash, then that milk watered down to 8.2 would still have 0.70 per cent. of ash. Mr. Richmond would argue that this milk must be genuine, as the ash was not materially deficient; and yet it had been artificially brought down from normality to exceptional and rare poverty. Mr. Richmond's argument from the constancy of the proportion of milk-sugar in normal milk was no stronger. He stated that normally the milk-sugar amounted to 53 per cent. of the solids-not-fat, with a variation of 2 per cent. either way. Considering that the total amount of milk-sugar in milk was only 4 per cent., and that milk-sugar was the constituent most liable to rapid diminution, and that our methods of estimating milk-sugar were hardly so perfect that a variation of, say, 0.2 per cent. would be an inexcusable error in analysis, that amount being the whole of the balance between milk-sugar in genuine, poor, and watered adulterated milk, it appeared to him far from safe to found upon this circumstance such a superstructure as Mr. Richmond had raised.

It thus came all back to the nitrate test, which must give a reaction with a milk low in solids-not-fat before such milk could be safely condemned. But Mr. Richmond admitted that he could not find by that test 10 per cent. of London water. It must, lastly, not be forgotten that, unlike Mr. Richmond, who could follow his samples back to the cow, the Public Analyst was not in that fortunate position, and had solely to rely on his analytical figures in giving a certificate upon a sample of milk. Would Mr. Richmond, were he in the position of the Public Analyst, have made the suggestions which he had brought forward that evening? At the same time, the society was thankful to the author of the paper, which, if not at present very practicable, was at least eminently suggestive.

Mr. Richmond, in reply, said that he was glad to hear Mr. Hehner's full and frank criticism, as many of the points raised by Mr. Hehner had occurred to him, and he was perfectly well aware, in bringing this paper before the Society, that it was a question of small differences and very careful determinations. Mr. Hehner's first objection was that the size of the fat-globules was affected by the stage of lactation. He was aware that, speaking from limited experience, the size of the large globules was affected; but it was the proportion of the very small ones which were not removed by separating milk, and upon this he thought an opinion might be based. He brought forward the idea in the hope that someone possessing time, patience, and microscopical skill would take it up. As far as his experience went, the proportion of the very small to the large was not very variable, but he would not like to give a positive opinion on the point. A complete micrographical study of milk even if it did not give results analytically useful, would be of the greatest value. With reference to the question of "separator slime," he would point out that Mr. Faber had said that it contained all the dirt of the milk, but did not state that it

consisted wholly of dirt. He could not think that Mr. Hehner had examined this substance. Certainly there was a layer of dirt, but there was a white layer on the top of this, and of course it was possible—he might be wrong in what he had stated—that the white substance might also be dirt. He had not examined it very fully, but all his results pointed to the presence of a substance which was not dirt. He still thought that this determination might afford evidence of adulteration. With regard to the evidence afforded by milk-sugar estimations—and this would also reply to Mr. Bodmer's question as to the determination of this substance—he distinctly stated that he did not lay very great stress upon it, chiefly because the methods of estimating the milk-sugar and proteids were not very exact; it was merely a corroborative test, but he did not think that it should be utterly rejected on that account. He knew that the difference of two-tenths of a per cent. was almost within the limits of experimental error, but he thought that with careful working, and if the sugar were determined by the polariscope, closer results might be attained; he did not think that it could be worked by other methods as accurately as with the polariscope. In doing his sugar determinations he exactly followed Dr. Vieth's procedure, with the exception that he used a correction for the polariscope, which Dr. Vieth had not done. Mr. Hehner's chief point against the evidence afforded by the ash was that if a milk contained 8.5 of solids-not-fat, and 0.73 of ash, and it was watered down to 8.2, the ash would be found to be 0.70. He (Mr. Richmond) would call 0.73 a high ash in a milk containing 8.5 per cent. solids-not-fat. He thought that the larger number of milks having 8.5 per cent. of solids-not-fat would contain less than that, and nearer 0.70 per cent. He would not pretend that he had come forward with methods which would absolutely detect every case of milk-adulteration, no matter what proportion of water was contained; but as all the evidence went to show that abnormally low milks contained a high ash, an analyst who found a milk with low solids-not-fat and low ash could give evidence that it had not the composition of an abnormal milk, should this be suggested by the defence. When estimating the ash he sometimes used an argand burner, and sometimes a muffle. He thought that the muffle was a piece of apparatus which was not sufficiently used in chemical laboratories, for by its means almost any temperature could be obtained. It was possible to ignite with a muffle at a very low temperature.

He had also used an argand burner when examining single samples, and in that case, as he had been very careful to avoid a red heat, he did not think his ash figures were under-estimated. He hardly ever found a difference of more than 0.02 per cent. in duplicate estimations. He had not intended to say that, by the proportion of ash to the solids-not-fat, all cases of watering could be detected, but he still believed that it would be a valuable determination in doubtful cases. He had already explained that the ratio of solids-not-fat to ash was, approximately, 100 to 8.3.

What he intended to say in connection with the farmer watering the milk was, that in the evidence given in courts the blame was always put upon the farmer, and this evidence would not be entirely untrue.

In reply to Mr. Cassal's question as to whether, supposing that a milk had been adulterated to the extent of 10 per cent. with London water containing 0.18 parts

per hundred thousand of nitrogen as nitrates, that amount could be detected by his process, he had not tried the experiment with London water, but should think that it was possible that the test might fail.

Mr. Bevan's observation that the nitrates might occur in milk, differed from his own, but he had no hesitation in accepting the statement. He would not like to indulge in any theories as to whether the cow had drunk water containing nitrates, or whether it had eaten roots, or anything else. All the milks which he had examined came from farms in which the water was good, and the quantity of nitric acid was extremely small.

[NOTE.—Since reading the paper I have had given to three cows one gramme of potassium nitrate per day in their food for three days; eight hours after the last dose of potassium nitrate was administered, the cows were milked, and the milk gave a strong reaction for nitric acid; twenty hours after the last dose the cows were again milked, and the reaction for nitric acid, though not so strong, was quite distinct. As I have on several occasions found on farms waters containing from 10 to 15 parts per 100,000 of nitric acid, a quantity equal to one gramme of potassium nitrate might easily be absorbed daily by the cattle. Mr. Bevan's observation and my own render it necessary to discard the "nitric acid test" for water in milk.—H. D. R.]

The remaining paper was :

ON THE EXAMINATION OF COPPER SULPHATE.

By JOHN RUFFLE, F.I.C., ETC.

THE increasing employment of copper sulphate as a vine-dressing for the phylloxera, and as a preventive dressing for potato disease, directs more attention to the analysis of commercial copper sulphate.

The usual good qualities, as found in the market, are sold on a guarantee of 98 per cent., and contain only traces of iron and other metallic impurities; the lacking 2 per cent. is made up by a little free sulphuric acid from the mother liquor, which remains imprisoned between the crystals, and moisture left behind through a more or less incomplete drying of the crystals after washing.

To arrive at the true amount of copper sulphate, take 1 to 2 grammes, dissolve in water, neutralize with ammonia, make up to from 300 c.c. to 400 c.c. with water, and acidulate with about 3 grammes of sulphuric acid, adding this, of course, in a diluted state. Turn on a steady electrical current from any battery, but of such strength as will give 20 c.c. to 50 c.c. of electrolytic gas per hour, employing platinum anode and cathode. Leave over-night, wash cathode with water and absolute alcohol, dry, cool, and weigh. The larger the cathode surface, within reasonable limits, the better, a brighter and more compact deposit being thus obtained. A total cathode area of about 20 square inches answers well for the deposition of up to 0.5 gramme of Cu. Calculate the Cu obtained into $\text{CuSO}_4\cdot 5\text{H}_2\text{O}$.

For the free acid, take 20 to 50 grammes of the crushed crystals, stir well with successive portions of absolute alcohol, add water to the alcoholic extract, a few drops

of phenol-phathlein solution, titrate with standard soda, and calculate into free sulphuric acid.

The determination of the excess of water or "moisture," though at first sight apparently an easy operation, is not really so, and cannot be done by any one known method. Experiments made to discover the readiest way of estimating the "moisture," show that :

First, the true amount of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ present must be found.

Second, a portion must be dried at a temperature of 100°C ., to constant weight, and the loss noted.

To effect this second point, take 3 to 5 grammes of the crushed sample, expose to 100°C . for two hours, cool, and weigh. The loss will be the excess of free water, plus the amount of water of crystallization due to four out of the five molecules of water in the true amount of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ present.

The loss of $4\text{H}_2\text{O}$ at 100°C . in pure 100 per cent. $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ is 28.91 per cent.* If the percentage be not 100, but only 98, the loss therefrom would then be only 28.33 per cent., and if the total loss at 100°C . were, say, 29.50 per cent., this, minus the 28.33, would leave 1.17 per cent. difference as the amount of "moisture."

This procedure of determining the $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, as well as drying at 100°C ., is an unavoidable double operation, since experiments show that no known method can be employed to effect the "moisture" determination without encroaching more or less upon the four molecules of water of crystallization. These seem to be held very loosely, whilst the fifth is retained with great tenacity.

Two examples of excellent high-class copper sulphate, treated as above, will show the results obtained, the one being a sample of wet, undried crystals, the other of crystals dry and ready for casking :

	Wet.		Dry.
Copper found by electrolysis	24.52	24.90
Total loss at 100°C	30.60	29.42
Less that due to $4\text{H}_2\text{O}$ at 96.94 per cent....	28.31	at 98.42 per cent.	28.45
	2.29		.97

giving :

	Wet.		Dry.
Crystals $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	96.94	98.42
Moisture	2.29	0.97
Free H_2SO_4	0.68	0.49
Undetermined and loss	0.09	0.12
	100.00		100.00

If examined minutely, the copper sulphate crystals will be found to contain traces of many of the metallic impurities which existed in the copper originally employed ; the principal of these is generally iron, but if the copper were auriferous or argentiferous, the gold or silver would be respectively detected in the crystals.

As illustrating the difficulty found in estimating the "moisture" alone, some

* Taking Cu = 63, O = 16, S = 32, H = 1.

experiments made may be quoted. Two portions of each of the foregoing "wet" and "dry" samples were exposed to the drying action of strong sulphuric acid under an air-pump, and the loss of weight incurred noted until constant. Constancy was reached in about six weeks, the final loss in each case being :

• Loss over sulphuric acid under air-pump :

						Dry.		Wet.
1st sample	30·22	...	29·43
2nd sample	30·31	...	29·38

The same was done in presence of freshly-fused calcium chloride, in about the same time.

Loss over fused calcium chloride under air-pump :

						Wet.		Dry.
1st sample	29·83	...	29·20
2nd sample	29·83	...	29·23

Dehydrated copper sulphate, dry sodium chloride and dry sodium nitrate tried in a similar manner all gave imperfect results.

It is interesting to notice and contrast the total loss at 100° C. with that over the sulphuric acid and calcium chloride, both being at the ordinary temperature of the air.

				Loss at 100° C.		Loss over H ₂ SO ₄ .		Loss over CaCl ₂ .
"Wet"	30·60	...	30·22	...	29·83
						30·31		29·83
"Dry"	29·42	...	29·38	...	29·20
						29·38		29·23

For the low qualities of crystals, it is only necessary to determine the copper, and calculate this into $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

Dr. Sykes exhibited a small gas boiling burner, which had been forwarded to him in his official capacity by Messrs. Fletcher and Co., of Warrington. It was intended to illustrate a method of enamelling recently introduced by that firm, who state that the covering applied will withstand exposure to a red heat for any length of time, that it does not chip off, and that it is practically indestructible. The enamel can be applied in any shade, colour, or variety of colours. He (the speaker) suggested that a very useful application of this invention would be the enamelling of the iron parts of apparatus used in the laboratory, for, with the varnish at present employed, such parts became very unsightly after being in use for some time.

(Conclusion of the Society's proceedings.)

The Analysis of Sperm-oil containing Mineral-oil. C. A. L. de Bruyn (*Chem. Zeit.*, 1893, xvii. 1453).—The rough qualitative test of the presence of mineral-oil in sperm-oil—consisting in saponifying a small quantity with alcoholic potash, diluting the liquid with water, and observing the separation of unsaponified mineral-oil—is not interfered with by the presence of cetyl alcohol and its congeners in normal sperm-oil, as these bodies are retained in suspension in the soap solution and only separate on standing. The accurate determination of mineral-oil in an adulterated

sample of sperm-oil can be effected by the usual method of saponification, followed by extraction of the soap solution with an immiscible solvent, and treatment of the resulting mixture of higher alcohols and mineral-oil with acetic anhydride. In carrying out this separation, the mixture of mineral-oil with higher alcohols is boiled for half an hour with double its volume of acetic anhydride. Complete solution takes place, but the mineral-oil separates again on cooling. The two layers are separated and the mineral-oil freed from acetic anhydride by washing with dilute alkali. The higher alcohols, which are obtained as esters on adding water to their solution in acetic anhydride, can be recovered and their amount determined.

The author points out that it would be desirable to know whether the percentage of higher alcohols is sensibly constant in pure sperm-oil. B. B.

NOTE BY ABTRACTOR.—The abstractor has found a nearly constant percentage of 40 per cent. of higher alcohols in pure sperm-oil; variations are often due to incomplete extraction rather than difference in composition.

The Acidimetric estimation of Potassium Borofluoride. F. Stolba. (*Casopis pro prumysl chemicky*, 1893, iii. 298; through *Chem. Zeit.*)—The principle of the method consists in the decomposition of potassium borofluoride by fusion with sodium carbonate according to the equation:



A weighed quantity of the borofluoride in a finely-powdered state is heated in a deep platinum crucible with about four times its weight of sodium bicarbonate, which is preferable to the carbonate, as it can be weighed more exactly. The quantity of sodium bicarbonate taken must be accurately known, as the estimation of the borofluoride is effected by means of the loss of alkalinity of the melt. When fusion is complete, and the escape of carbon dioxide has ceased, the contents of the crucible are dissolved in water in a capacious platinum dish, and the solution heated to boiling and titrated, using litmus as an indicator, and taking care to avoid the addition of much acid at one time, lest the borofluoride be re-formed. The presence of boric acid is said not to interfere. The alkalinity of the bicarbonate used must, of course, be determined.

B. B.

Critical Examination of Methods for the Estimation of Sulphur. L. Marchlewski. (*Zeit. anal. Chem.*, 1893, xxxii. 403; through *Chem. Zeit.*)—Gröger's method, which consists in heating the pyrites with metallic iron to convert it into ferrous sulphide, treating the latter with hydrochloric acid in a current of carbon dioxide, and absorbing the hydrogen sulphide in a solution of iodine of known titre, is inadmissible because any sulphates which may accompany the mineral are partially reduced to sulphides during the ignition with iron. With pure sulphides the method gives good results; for sulphates it is useless, inasmuch as reduction is never complete. Sauer determines the sulphur by burning the pyrites in oxygen, passing the sulphur dioxide into bromine water, and determining the sulphuric acid there produced; this can be recommended as far as the agreement of results is concerned, but is troublesome, and must give place to Lunge's method unless soluble sulphates be

present. Föhr's method, wherein the hydrogen sulphide obtained by suitably decomposing the mineral is absorbed in an ammoniacal solution of zinc sulphate, ferric sulphate (and presumably acid) added to the liquid containing the zinc sulphide, and the ferrous sulphate produced titrated with potassium permanganate, is not capable of yielding accurate results. Klobukow also recommends evolution of the sulphur as H_2S , using nascent hydrogen for the purpose, and absorbing this in iodine solution of known titre; this process is inadmissible, on the same grounds as those which render Gröger's method useless.

A. G. B.

The Oxidation of Fatty Oils. W. Fahrion. (*Chem. Zeit.*, 1893, xvii. 1453, 1454).—Sundry attempts have been made from time to time to determine directly the increase in weight of fatty oils allowed to oxidize in the air; various plans to expedite the rate of oxidation having been adopted. Thus Livache carried out experiments in which the oil was allowed to fall on finely-divided lead; while Vogel, more than thirty years ago, proposed cotton-wool as a support for the oil. The author has found that chamois-leather is more suitable for the purpose than either of the materials mentioned. It is, however, somewhat hygroscopic, and a blank experiment must be carried out at the same time as those with the oils, to correct for its changing content of water as nearly as possible.

Oil.	Iodine number.	Days' exposure.	Maximum gain.
Olive ...	82.1	42	0.2%
Sesame ...	110.2	21	3.6
Rape ...	102.4	10	2.8
Cotton-seed ...	109.2	10	5.6
Poppy ...	135.9	7	8.4
Walnut ...	149.2	6	9.0
Linseed ...	175.8	6	12.4
Cod ...	171.0	5	9.5

The number of days' exposure is the length of the period that elapsed before the full gain in the weight of the oils under test took place. A longer exposure does not necessarily increase the weight of an oil suffering oxidation, as volatile products, such as carbonic, formic, and acetic acids, may be formed. It is worthy of note that the iodine absorption of an oil does not give an accurate estimate of its power of absorbing oxygen. This becomes evident on converting the iodine numbers given above into their oxygen equivalents, and comparing the figures thus obtained with the oxygen absorbed, determined directly. This is due to the fact that, instead of the mere addition of an oxygen atom to an unsaturated compound, the insertion of an atom between adjacent atoms of carbon and hydrogen and the formation of an hydroxyl group may take place. Moreover, oleic acid, which absorbs iodine, does not readily take up oxygen, so that a discrepancy between the iodine and oxygen absorbed will be apparent in all oils containing oleic acid.

B. B.

Reactions of Hydrazines with Lignin. E. Nickel. (*Chem. Zeit.*, 1893, xvii. 1209 and 1243).—Phenylhydrazine hydrochloride may be used as a reagent for lignin, the aldehydic nature of which has been demonstrated, as follows: In an aqueous solution of phenylhydrazine hydrochloride wood acquires a light yellow

colour, which becomes deeper on addition of dilute hydrochloric acid (about 15 per cent.). When the sample is set aside the yellow colour changes to a pure green in the course of an hour, or longer.

Hydrazine sulphate (now obtainable from Kahlbaum) gives a very similar reaction with wood. In a cold solution of this salt the wood becomes yellow in a few minutes, the depth of colour depending on the nature of the wood, and attaining its maximum with bamboo-cane, among the woods which the author has examined. The subsequent action of 20 per cent. hydrochloric acid on the wood changes the yellow to orange.

It is characteristic of aromatic aldehydes to give colour-reactions with the hydrazines, and the above behaviour of wood might be due to the aromatic aldehydes which it is said to contain, namely, vanillin and cinnamyl aldehyde. An aqueous alcoholic solution of vanillin yields a yellow colour with hydrazine sulphate, and, on the addition of hydrochloric acid, a yellow precipitate falls, but there is no orange colour produced. The author has already denied that these aldehydes are constituents of wood, and he regards the above reaction of vanillin as confirmatory of his views.

A. G. B.

Valuation of Leather Glue. F. Gantter. (*Zeit. Anal. Chem.*, 1893, xxiix., 413; through *Chem. Zeit.*)—One hundred grammes of the shredded sample are heated with 1 litre of water containing a few drops of caustic soda solution until solution is complete, when the volume of the liquid is made up to 2 litres. After the solution has been set aside for ten hours, 20 c.c. (= 1 gramme of glue) of the clear liquid are evaporated, and the residue is dried at 105°, weighed and ashed. The weight of the ash-free raw glue is thus ascertained.

To estimate the pure glue in the sample, 20 c.c. of the above solution are transferred to a 100 c.c. cylinder, diluted with 30 c.c. of water, and neutralised with acetic acid. Tannin solution is then added, until no further precipitation occurs; the solution is shaken, made up to the mark with water, and filtered through a dry filter. The filtrate is shaken with hide powder, and set aside for ten hours to ensure complete elimination of tannin. After another filtration 50 c.c. of the solution are evaporated, and the residue dried, weighed, and ashed. By subtracting the weight of this residue, less that of the ash, from the weight of the ash-free raw glue, the percentage of pure glue substance is ascertained.

A. G. B.

Butter Distinguished from Margarine. F. Gantter. (*Zeit. Anal. Chem.*, 1893, xxxii. 411; through *Chem. Zeit.*)—The frequent occurrence of earth-nut oil in margarine renders the identification of the latter in butter an easy matter. Butter containing so little as 1 per cent. of earth-nut oil will give a dark brown-red colour when treated with concentrated sulphuric acid (ANALYST xviii. 183); pure butter should give a straw-yellow or reddish-yellow colour. The iodine number of butter-fat, as determined by the author's method (employment of a chloroform solution of iodine instead of Hübl's solution) should be between 13 and 16; that of earth-nut oil is 40-51.

A. G. B.

THE ANALYST.

DECEMBER, 1893.

THE LATE ROBERT HIGGINS DAVIES, F.I.C., F.C.S.

It is with the deepest regret that we have to record the death, at the early age of forty-two, of our esteemed colleague, Mr. R. H. Davies, one of the Secretaries to the Society of Public Analysts. He passed away, after a long illness, at Bournemouth, on November 16.

Mr. Davies was a native of Newport, Monmouthshire, and received his early education at Wesley College, Taunton, subsequently being articled as pupil to the late Mr. Edwards, of Dartford, Kent. He obtained, in 1871, the first of the three Junior Bell Scholarships, and became a student at the School of the Pharmaceutical Society, where his career was a distinguished one, for during this period he took, in 1872, silver medals (the highest award then given) in chemistry, botany, and materia medica, and a bronze medal for practical chemistry. On leaving the Pharmaceutical Society's School, he received an appointment from a leading firm in Dublin; this he held for a year, after which he became demonstrator in the laboratories of the Pharmaceutical Society. He also occupied for some time the position of private assistant to Dr. Attfield. Shortly afterwards he was chosen as Assistant Secretary to the Pharmaceutical Conference. He resigned his position as demonstrator on being appointed Public Analyst for Hammersmith, and afterwards was elected to the important office of Chemist and General Superintendent of the Drug Department of the Society of Apothecaries. In 1889 he was appointed a member of the Board of Examiners of the Pharmaceutical Society. These appointments he held up to his decease. He also served for three years on the council of the Institute of Chemistry.

As Mr. Davies' work lay principally in the direction of pharmacy, his papers, of which the following is a list, appeared in the *Journal of the Pharmaceutical Society*: "Use of Potassium Ferro- and Ferri-cyanides for the Detection of Cobalt and Nickel," 1876; "Constituents of the Ivy," 1876 and 1877; "Notes on Sulphate of Quinine," 1876; "Notes on Three New Chinese Fixed Oils," 1885; "The Iodine Absorptions of the Essential Oils," 1889. Besides these, the following joint papers appeared: "On the Composition of Easton's Syrup," by Davies and Schmidt, in 1883; "Notes on Eucalyptus Oils," by Davies and Pearmain, in 1891; and "Notes on Eucalyptol," by the same authors, in 1892. Many of his other papers have not been published.

Mr. Davies became a member of the Society of Public Analysts in 1887, was elected a member of council in 1889, and appointed one of its secretaries in 1891. He has also been a member of the Publication Committee of *THE ANALYST* since the *Journal* became the property of the Society.

In Mr. Davies the Society loses a valued member, one whose courteous and affable disposition had endeared him to the hearts of all his fellow members, whilst his sound and practical judgment gave considerable weight to his expressed opinions. His loss will be deeply felt by a wide circle of friends both in and outside the Society. He leaves a widow and two sons.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on November 1st, at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner occupied the chair

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed as members: J. Kear Colwell, F.I.C., 101, Great Russell Street, W.C., and H. H. B. Shepherd, F.I.C., Northcote, Mount Pleasant Lane, Upper Clapton, N.E.

Mr. George Rudd Thompson, F.I.C., Newport, Mon., was elected a member of the Society.

The following paper was read by its author:

NOTE ON AN ABNORMAL MELTING-POINT.

By E. J. BEVAN, F.I.C.

In the course of an examination of a crystalline fatty body obtained from pure lard by fractional crystallization from ether, I had occasion to determine its melting-point. For this purpose capillary tubes were filled with the melted body in the usual way. The melting-point, determined within a few minutes of filling the tube, was 47° C. Another tube, filled at the same time, but examined the next day, gave a melting-point of 61.5° . This tube, after allowing the body to solidify, was plunged into water at 50° , and, to my great surprise, was found to melt instantly to a perfectly clear fluid. The experiment was repeated at intervals down to a temperature of 45.5° , at which point it remained solid. A number of experiments were made on a larger scale, using test-tubes internally coated. After standing twenty-four hours, the substance remained solid, when the tube was plunged into water at 50° . One half of the contents of the tube was then melted over a burner and allowed to solidify.

On plunging the tube into water at 50° it was found that the portion recently heated melted to a clear liquid, the remainder being quite solid and opaque; and the portion melted corresponded exactly with the part of the tube heated.

It is clear, therefore, that the body has two very definite and widely different melting-points, and that it passes slowly and spontaneously from the state of low melting-point to that of the higher, and can be made to pass from the high to the lower state immediately by simple heating.

As I hope to show you, it can also be made to pass from the lower to the higher state by gradually increasing the temperature.

Thus, I take a tube recently heated and place it in water at about 43° . At this

temperature it remains solid; on gradually raising the temperature, I find that the substance melts to a clear liquid at about 47°. If now the temperature be still further gradually increased, the substance passes again into the solid state at about 53°, and does not melt again till a temperature of about 62° is reached.

The existence of the two melting-points was confirmed by the use of Cross and Bevan's method, as described in the *Chem. Soc. Journal* for 1882.

These experiments emphasize the importance, which has before been insisted on, of delaying the determination of melting-points for some hours after the substance has been melted.

It has since been pointed out to me by Mr. Allen that some curious experiments by Mr. T. Maltby Clague on the alteration of the melting-point of cocoa-butter, are recorded on p. 570 of his *Commercial Organic Analysis*, vol. iii., part ii.

The Chairman (Mr. Otto Hehner) remarked that the subject was very important. Analysts often met with such difficulties in analyzing fatty substances. It would be very desirable to come to some agreement as to the conditions under which the melting-point of a fat ought to be taken.

In the absence of Mr. Stokes, the Secretary (Dr. Dyer) ready the following paper:

ON THE FORM OF CERTIFICATES ON ADULTERATED SAMPLES.

By A. W. STOKES, F.I.C.

Finding a sample of milk submitted to me by St. Luke's Vestry to be deprived of 25 per cent. of its cream, I certified to this in the following way on the legal form:

"I am of opinion that the said sample contained the parts as under, or the percentages of* ingredients as under:

"Twenty-five per cent. less than the normal proportion of cream.

"Observations.

"No change had taken place in the constitution of the sample that would interfere with the analysis."

The Magistrate ruled that as I did not give the component parts of the milk, the certificate was invalid. The Vestry laid the facts before the Local Government Board. They replied as follows:

"Local Government Board,
"Whitehall, S.W.,
"10th October, 1898.

"SIR,—I am directed by the Local Government Board to state that they have had under consideration your letter of the 22nd ultimo in reference to a difficulty which has arisen respecting an analyst's certificate under the Sale of Food and Drugs Act, 1875, as to a sample of milk.

"The Board have also had before them a copy of the certificate referred to, which was enclosed in your letter of the 27th ultimo.

"I am now to state that it appears to the Board that the difficulty has arisen, not from the provisions of the Act, but from the particular form of words adopted by the analyst in the case in question. They consider that in the form of certificate

* The word "foreign" in the certificate standing before "ingredients" was cancelled in ink.

prescribed by the Act the analyst is required either to state 'the parts' contained in the sample or 'the percentage of foreign ingredients,' but that there is no obligation on him to state both the parts and the foreign ingredients, one requirement being alternative to the other. They do not understand, therefore, why in the case in question the analyst should not have stated in his certificate that the sample contained 'the parts as under,' viz., so many parts of fat, so many parts of water. Having done this, his proper course would apparently have been to enter, under the heading 'Observations' in the prescribed form, that he was of opinion that this statement of 'parts' showed that abstraction of a portion of the milk (*i.e.*, 25 per cent. of its cream) had taken place.

"The Board agree in the view that the certificate as it stands is invalid, since it neither gives the 'parts' nor the 'percentages of foreign ingredients.'

"I am, sir, your obedient servant,

"C. N. DALTON,

"Assistant Secretary.

"G. W. PRESTON, Esq.,

"Clerk to the Vestry of the

"Parish of St. Luke, Middlesex."

I cannot see that the view taken by the Local Government Board can possibly comply with the Act. A reference to the schedule of the Sale of Food and Drugs Act, 1875, shows that under the word "Observations" directions are given thus: "Here the analyst may insert at his discretion his opinion as to whether the mixture (if any) was for the purpose of rendering the article portable or palatable, or of preserving it or of improving the appearance, or was unavoidable, and may state whether in excess of what is ordinary, or otherwise, and whether the ingredients or materials mixed are or are not injurious to health. In the case of a certificate regarding milk, butter, or any article liable to decomposition, the analyst shall specially report whether any change had taken place in the constitution of the article that would interfere with the analysis."

These precise directions as to the use of the "Observations" column seem quite to preclude placing there the amount of the adulteration. It seemed to me that in placing the words "25 per cent. less than the normal proportion of cream" where I did, I was stating "the parts as under," so far as the adulteration was concerned. I thought that the adulteration form was intended to show the extent of the adulteration of the sample, and not to give the full component parts of the sample. Otherwise the "genuine" form might equally have filled into it the component parts of the article. Knowing the interest of the subject to so many members of the Society, I venture to trouble you with this note, so that those concerned may see the view taken by some authorities on the subject, and may act accordingly.

DISCUSSION.

Dr. Bernard Dyer said that the Local Government Board had informed Mr. Stokes in their communication that his certificate was irregular, inasmuch as he had not stated "parts." He (Dr. Dyer) thought it was impossible to state parts which were not there. Mr. Stokes's proper course would have been to say that the sample contained 100 parts of skimmed milk, and, in his observations, to state the extent of the skimming.

Mr. Hehner thought that the correspondence had convinced the members who

were present that quibbling was not confined strictly to the members of the legal profession; but, in view of the possibility of a very long discussion on the subject—in which every public analyst took some interest—he trusted that members would contribute towards the enlightenment of Mr. Stokes, and not protract the proceedings at the expense of the vinegar question.

Mr. Alfred H. Allen said that the magistrates acting in one of his districts, at the suggestion of the solicitor for the defence, had held that the whole of the analysis of a milk ought to be given. On another occasion the same Bench held that, where soda-water containing lead was in question, he ought to give the percentage of lead, not the grains per gallon or per 100,000, and the full analysis of the water containing it. In the case of milk, he was in the habit of stating, "The sample contains the parts as under: milk 88, added water 12 per cent., total 100"; and he could not see how, if the prosecution was properly conducted, the defendant's solicitor could get over that. In the case of skimmed milk, he thought analysts were bound to give the constituents, if from the results of the analysis an opinion was formed that the sample had been skimmed, or was deficient in fat. It might happen in the future that a method of analyzing milk would be devised which was entirely independent of a determination of total solids, direct or indirect; and if that should happen, was an analyst to be compelled to give analytical data which he did not require for the purpose of forming his own opinion on the sample? In the case of butter, coffee, and other articles, the contention that the analytical data should be given was still more preposterous, as only an expert could interpret them.

Mr. Cassal remarked that he had read a paper before the Society in which he had dealt with the point raised by Mr. Stokes. He should be glad if the Local Government Board could see their way to give public analysts a little more active support, and would give some further consideration to the difficulties by which public analysts were beset. He had just perused the letter. Objection might certainly be taken to the form of this certificate, because it neither gave "percentage of foreign ingredients" nor "parts as under"; but while these words were suggested in the schedule, they were not compulsory under the Act. He (Mr. Cassal) had had occasion to point out to the Society that it was perfectly possible, in the case of fat abstraction from milk, to give "parts as under." When this was done, the certificate was strictly in accordance with the terms of the schedule. In this case there was 25 per cent. of fat abstracted. Mr. Stokes had certified that there was "25 per cent. less than the normal proportion of *cream*," a form of statement which was not one which he (Mr. Cassal) thought desirable, and which could hardly be regarded as accurate, since cream was not fat; but it was perfectly possible to conform with the suggestions of the schedule, and to use the words "parts as under" by saying that "the said sample contained the parts as under," so many parts of milk of genuine composition, but of the poorest quality, and so many parts of milk devoid of fat. It could not be said that that was not the composition of the article. Where there was no added water, the percentage of fat abstracted was obviously the same figure as the percentage of milk devoid of fat. This form of certificate gave the actual composition as determined by analysis of the sample submitted to the analyst. There could be no quibble raised upon that form of certificate, because it was strictly in

the form suggested by the schedule, utterly foolish as that form was, and because it showed the actual composition of the sample, which was all the Court and the public required to know. The public had nothing to do with the data upon which an analyst formed his opinion. There was nothing in the Act which required a public analyst to state analytical details. He had thought that the members of the Society were pretty well agreed some time ago that it was most undesirable that public analysts should state analytical data in their certificates. He (Mr. Cassal) sincerely hoped that the Society would adhere to this position, and that, as far as possible, all analytical data would be kept out of reports, certificates and legal cases.

Mr. E. J. Bevan suggested that the discussion be postponed to the next meeting. It was a matter which very closely affected public analysts, especially those in the neighbourhood of London. He could mention one or two curious and interesting circumstances in connection with the wording of certificates, and no doubt other public analysts would like to do the same.

Dr. W. Morgan wished to know if he was to understand that Mr. Cassal maintained that no percentages should be given in certificates. He believed analysts were bound to give the "parts as under," or the percentages of foreign ingredients. His practice, in the case of skimmed milk, was to say that 100 parts of the sample contained so much genuine whole milk and so much skimmed milk. He had had a peculiar case in which he certified the presence of an aniline dye in jam, but it was dismissed because he had not given the percentages.

Mr. Cassal observed that Dr. Morgan did not differ from him to any appreciable extent. Dr. Morgan was really advocating practically the form of certificate which he (Mr. Cassal) had previously mentioned that evening, where, under the words "parts as under," so many parts of milk of genuine composition and so many parts of milk devoid of fat were stated. If Dr. Morgan considered that the skimmed milk he referred to was devoid of fat, he (Mr. Cassal) inferred that there was no difference between their views.

Mr. Bevan argued that, as the schedule stated that the "parts as under," or words "to the like effect," were to be given, clearly the words "to the like effect" embraced the case mentioned by Mr. Stokes. Mr. Haden Corser had objected to his (Mr. Bevan's) certificates, which were usually in this form: "I am of opinion that the sample contains 10 per cent. of added water"; and, at the solicitation of the Clerk to the Board, the certificate was altered thus: "I am of opinion that the sample contains, milk 90 per cent., added water 10 per cent." As the Act allowed analysts to use words "to the like effect," this was clearly an allowable form of certificate. Moreover, the schedule of the Act stated that the percentages of foreign ingredients might be given. If this were done, he thought that, on the other hand, a simple statement of the percentage of the ingredients taken out should be sufficient.

Mr. Hehner considered that it was very unadvisable to give a certificate which did not strictly and literally comply with the schedules given in the Act when such a certificate could easily have been given, and, although the Local Government

Board's letter was not clearly worded, still, it contained a fair, if not a sufficiently explicit, suggestion. He thought there could not be much difficulty at the present time, especially for an analyst as experienced in the working of the Sale of Food and Drugs Act, as Mr. Stokes undoubtedly was, to word certificates so that they absolutely complied with the Act. Mr. Stokes seemed to be in dread of the opinion of some individuals, particularly solicitors, or possibly magistrates. It never entered into his—Mr. Hehner's—mind to trouble himself about what a magistrate or a solicitor might think, provided he complied with the Act. Nothing could prevent a bench of magistrates from dismissing a case, however accurate the certificate might be. He had for some time past made it a rule to omit in every case giving in his certificates any semblance of an analytical figure, but to state what were the "parts as under"; and he had never found that there had been any serious difficulty. He had often been asked to supply analytical figures, but, whilst he was perfectly willing to give them in a letter, he would not do so in a certificate. Analytical figures were absolutely unintelligible to anybody but an analyst. A public analyst obtained his figures, not for the information of other people, but for his own use—to guide him in giving his opinion under the Act, or, in other words, to enable him to arrive at a correct opinion. The analytical figures were simply the means to satisfy himself, and to assist him in forming his opinion. It seemed preposterous that a bench of persons not scientifically educated should be allowed to judge of analytical figures at all; and he also thought that an analyst should not show on his certificate the means that he used in order to enable him to give his opinion. If he were asked to give his reasons in court, then he would be bound to give them, and also his figures, and he would be glad enough to do so, because he would know more about the figures than anybody who might be in court could know. He was aware that the Act said "words to the like effect," but, unfortunately, the use of "words to the like effect" had led to the dismissal of cases. It would require a decision of the High Court to decide what were "words to the like effect." It was desirable that there should be such words in reference to such cases, for instance, as those of lead in soda-water, where the lead was expressed in grains per gallon; or in that of alum in bread, where the alum was expressed as so many grains in a 4-lb. loaf. He hoped that members of the Society would adhere as strictly as possible to the schedule of the Act, whether they thought it unreasonable or not. It was best to comply with the suggested form. Analysts did comply with it when they gave the percentages of foreign ingredients in the case of adulterated articles; and he did not think that any chemists need trouble themselves about the matter, even if gentlemen like Mr. Ricketts asked for more.

A long discussion on the vinegar question now followed, the report of which will appear in our next issue.

(Conclusion of the Society's proceedings.)

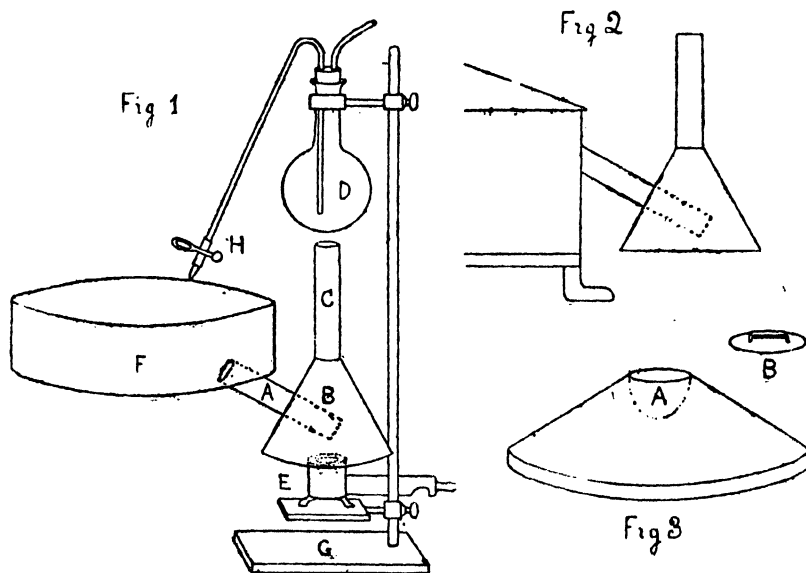
NEW FORM OF APPARATUS FOR HEATING THE WATER IN CENTRIFUGAL MILK-TESTERS.

By ROBERT H. WILKINSON.

ALL who have used the Babcock milk-tester as at present constructed will have found a difficulty in keeping the machine at a proper temperature, and the clumsy method of adding hot water to the test-bottles, recommended by the makers of the machine, will not find favour in the eyes of any owner of a "wash-bottle."

For the past two years I have had in constant use two 25-bottle-power machines, sometimes making as many as 200 estimations of fat per diem. Under these circumstances the necessity of changing the water frequently was particularly tiresome. By a very simple contrivance I have overcome all the difficulties as regards heating the water required for filling up the test-bottles, and also for heating the water in the tank of the machine.

A glance at the accompanying sketch will show how this is accomplished.



A hole is cut in the bottom of the tank F, and a metal tube, A, closed at the lower end, is fixed to the tank.

The tube A is surrounded at the lower or outer end by a metal shield, in shape like an inverted funnel, B, the stem of which, C, is continued above the highest part of the machine.

The mode of working is as follows: Cold water is poured into the tank F until tube A is filled, and the bottom of the tank covered to the depth of one-eighth of an inch. A wash-bottle, D, provided with a long flexible outlet tube with a glass jet and clip at the end, is clamped in position on the top of C. The burner E is now lit, and

the tank F covered. The test-bottles are now filled with milk and acid, and fixed in their place in the machine, which by the time the bottles are ready is quite hot, and the water in G nearly boiling. After whirling, the bottles are readily filled with hot water by means of the syphon connected with the wash-bottle, the current being controlled by the clip H.

I find a tube two inches by twelve quite large enough to heat a 25-bottle machine in twenty minutes when a Fletcher solid-flame burner is used. The smaller hand-machines would not require so large a burner or tube. The tube also, in machines where the driving-gear is at the side, could be conveniently fixed to the side of the tank F, and sufficiently high up to allow a burner to be placed under the lower end, as in Fig. 2.

Double-jacketing the cover of the machine, as described in the October ANALYST, means more trouble in changing the water in the jacket when it cools.

Precisely the same effect can be produced by using a cover having a cup-shaped depression in the lid A for hot water, with a suitable removable cover, B, Fig. 3.

Lansdowne Laboratory, Limerick.

WATER ANALYSIS—THE INTERPRETATION OF RESULTS.

By F. WALLIS STODDART.

(Reprinted from "The Practitioner.")

THE question of the utility of chemical analysis as a means of determining the wholesomeness or the reverse of potable water has lately been raised in several quarters; partly, no doubt, in consequence of outbreaks of enteric fever attributed with more or less reason to the use of water supplies not demonstrably polluted, but largely also as a result of the widely different opinions not unfrequently expressed as to the organic purity of one and the same water by different analysts.

In spite of all that has been written on the subject, no point in water analysis still remains so frequent a cause of this unfortunate variance of opinion as the meaning attributable to the presence of nitrates.

This is largely due to the fact that water analysis is to a great extent in the hands of persons who have little or no opportunity of thoroughly examining the question for themselves, and who, therefore, are necessarily guided by the published opinions of leading sanitarians.

Now, the text-book which is, and has been for years, used exclusively by the vast majority of teachers of chemistry takes the extreme view that nitrates in potable waters are absolutely without sanitary signification; the natural consequence being that there is a very large and annually increasing number of persons engaged more or less upon water analysis who, confessedly adopting this view without any modification, do not take the question of nitrates into consideration, and do not as a rule make any attempt to detect the presence of these salts.

On the other hand, there are numerous practical workers at this subject who

are convinced from long experience that the presence of nitrates in potable waters in proportions exceeding a certain minimum is a most important indication of pollution. Text-books which advocate this view are of course not wanting, but, for reasons into which it is not now necessary to go, they do not find a place in many educational establishments; whilst there are many treating more or less fully of water analysis which leave the reader practically in the dark.

It is surprising that a more critical examination of this question has not been published, as conflicts of opinion are naturally not uncommon, especially where action is taken under the Public Health Act. Now, however, that an important Government Department, popularly looked upon as a court of appeal in similar matters, has taken a course entirely opposed to the experience of almost all practical sanitarians (ANALYST, xviii. 246), thereby raising a serious obstacle to a much-needed sanitary improvement, it appears to me, in view of the present invasion of this country by a terribly fatal disease, disseminated almost invariably by means of drinking water, that it is of the utmost importance that this question should, if possible, be settled once for all. It is in the hope of contributing towards this desirable consummation that I venture to place before those interested in the maintenance of public health the following considerations, largely suggested by recent advances in our knowledge of the origin and mode of formation of nitrates (and nitrites) under the conditions naturally prevailing in the soil.

That nitrates in natural waters are, to quote the most recent work, "nearly exclusively derived from the decomposition of animal matters," is pretty generally recognised; though certain objections have been urged against this view, and will be discussed subsequently. It is contended, however, by one school of chemists that the presence of nitrates in whatever quantity, if unaccompanied by much unoxidized nitrogenous matter, is unobjectionable; by a second, that water containing more than a certain minimal quantity should be condemned as dangerous to health.

The latter class is further divided upon what may at first appear a merely theoretical point, namely, whether the danger attaching to the use of water of the character under discussion is of the same degree as that involved in drinking any sewage-polluted water, or is contingent upon the sewage matters, under slightly altered conditions, reaching the water in a less oxidized state. Those who hold the former view are probably comparatively few, but are, I believe, one and all practical sanitarians who have been forced to take it both by a careful consideration of what is known of the causation of disease, and by the repeated observation of obviously polluted waters, which, though they yield insignificant quantities of unoxidized nitrogen, in some cases, at least, have been reasonably credited with communicating disease on evidence in all respects similar to that which is generally accepted as establishing the connection between sewage pollution and the dissemination of cholera and typhoid. It may be of little consequence which view is adopted in advising a private client; but in taking legal steps to close wells on public grounds in the face of opposition, it is most important to come to a clear understanding on this point; and it will be well to bear this in mind whilst considering the broader question—Are we, or are we not, to ignore nitrates altogether?

It will facilitate the discussion of this matter if we first formulate the arguments which have been advanced by those who deny any sanitary significance to the presence of nitrates in drinking water, and then consider each in detail.

These arguments are as follows :

I. Nitrates in themselves, and in the proportions in which they occur in natural waters, are harmless.

II. Nitrates may be present in appreciable quantities in pure waters, and may be absent from polluted ones.

III. Nitrification being essentially a process of oxidation, objectionable matters are "burnt up."

IV. The completion of nitrification is a proof that the water has filtered through a layer of soil of such thickness that it is necessarily deprived of harmful impurities.

V. That there is a biological antagonism between the organisms effecting the various stages culminating in nitrification and those constituting the infective material, resulting in the destruction of the latter.

I. Now, the first argument is so obviously beside the question that it would seem to be a waste of time to consider it, were it not that the legal advocates in disputed cases make considerable capital out of it; and further, that it appears prominently in the water report which affords the motive for these remarks.

Plainly, the same statement applies with equal exactness to each of the other determinations made with the view of ascertaining the presence or absence of sewage; all such determinations being made with the object, not of detecting directly harmful matter, but of finding whether the character of the water is such that it may be the means of conveying infection. And yet it appears to have been overlooked that each grain of nitrogen in this form means very nearly six grains of calcium (or other) nitrate, and that the permanent hardness of the water is proportionately increased. It would appear, therefore, a matter worth consideration whether a water containing thirty grains of calcium or magnesium nitrate per gallon—a very usual amount—should be passed as perfectly wholesome.

II. The question of the natural occurrence of nitrates in undoubtedly pure—that is, dietetically satisfactory—waters is one that merits more attention. It is asserted that these salts occur naturally in such quantity as to render their determination meaningless for sanitary purposes.

Now, on looking into this argument one finds that it consists, first, of suggestions of innocent sources of nitrates; secondly, of instances of pure waters containing nitrates.

The possible sources of nitrates other than animal matters are :

- (a) Natural deposits of nitrates.
- (b) Ancient organic remains, or "fossil sewage."
- (c) Rain-water.
- (d) Vegetable matter.

(a) That there is or can be a deposit of nitrates in any geological formation in our climate is scarcely conceivable, and has certainly never been demonstrated. It

is established that nitrates are amongst the salts most easily washed out of the soil, and any accumulation in the more superficial strata—assuming that such could ever have been formed under different climatic conditions—must have been removed long before the era of water analysis.

Moreover, under the most frequently occurring conditions nitrates are not permanent, but speedily undergo decomposition under the influence partly of living organisms, partly of purely chemical agencies.

Further, if this assumption were true, it would naturally be anticipated that deep borings would yield water containing a higher proportion of nitrates than superficial wells. This is contrary, of course, to all experience. Nitrates are always found in greatest abundance at or near the surface, and in decreasing amount the further from the surface the point of supply is situated. I shall return to this point in connection with deep wells.

(b) The possible production of nitrates from the fossil remains abundant in some formations has been frequently asserted, but I have been unable to find anything like evidence in support.

The only systematic attempt to determine this question with which I am acquainted is an investigation by Mr. Chas. Ekin (*Journ. Chem. Soc.*, 1871, p. 64), in the course of which selected specimens of rocks and fossils, as well as spring waters from various formations, were examined, with the result that nitrogen was found in small quantities in material apparently out of reach of contamination. At the time, the view was expressed by Mr. Ekin that his results indicated that "the previous sewage contamination theory ought to be considerably modified."

Both the methods adopted and the deductions drawn appear to be open to criticism; but the need for any is dispelled by a subsequent contribution to the subject by the same writer, in which he says ("Potable Water," 2nd ed., 1880): "When the amount [of nitrogen as nitrates] exceeds .5 or .6 parts per 100,000, it points significantly to dangerous pollution;" and again: "This view of the importance to be attached to an abnormal quantity of nitrates has been formed in spite of a considerable predisposition to a contrary opinion, and has been literally forced upon the writer again and again by the investigation of cases which really leave no doubt in the matter."

The whole of this part of the question is obscured by a lingering remnant of the notion that any nitrogenous matter exposed to air may forthwith be directly oxidized with formation of nitrates; whereas the latter can practically only be formed in nature as the product of one of the steps of decomposition necessarily preceded by the earlier stages of putrefaction. To suppose, therefore, that nitrates can arise from the oxidation of fossils implies that the latter are capable of putrefaction; in which case our palæontological museums would require the intervention of the inspector of nuisances. Fossils may possibly retain traces of nitrogen; but if so these traces simply constitute a non-putrescible residue, and there is no valid reason to suppose that at this period of time there is any possibility of further change.

(c) The presence of nitrogen in rain-water in the shape of ammonia, nitrites,

and nitrates is a matter constantly coming under the notice of every water analyst and has been the subject of special investigation by many well-known chemists.

The Rivers Pollution Commissioners devote a considerable part of their sixth report to this question, and their results briefly amount to this: That rain-water collected with suitable precautions may contain nitrogen in this shape in quantities ranging from .0028 to .0875 grain per gallon, the average being .0238. This average quantity the Commissioners proposed to deduct from the nitrogen as nitrates, nitrites, and ammonia, found in natural waters, the remainder being attributed to animal matter. Thus rain-water may contain nearly one-tenth of a grain of nitrogen per gallon, the whole of which might be converted into nitric acid under suitable conditions. On the other hand, it is to be noted that it is, as a rule, only near towns that this maximum is approached, owing, probably, to the presence of ammonia produced by the combustion of coal; and that in these towns the storm-water does not generally pass into the soil, but is diverted into the drainage system; whilst it is extremely improbable that where rain falls upon land covered with vegetation, much, if any, of the nitrogenous compounds escape assimilation. This is corroborated by the fact that analyses of water from tracts of uncultivated land out of reach of animal contamination show often an absence, and never more than minute traces, of nitrates.

(d) It is the practice of those writers who disregard the presence of nitrates to refer to their derivation from "animal or vegetable organic matter" as if the distinction were of no importance. From a sanitary point of view, however, it is manifestly very important to determine, if possible, whether there is any distinction to be drawn between these two classes of organic matter in this respect. Now, the proportion of saline ammonia to albuminoid ammonia is well known to afford a practical means of distinction between animal and vegetable contamination in water.

Take, for example, the following instances of organic pollution, the results being stated in grains per gallon:

	A.	B.
Saline ammonia009	2.57
Albuminoid ammonia168	.14

A is a sample from a few inches of water remaining in an exhausted reservoir, the bottom of which is covered with a deposit of vegetable debris—not, however, entirely destitute of animal life, for it is naturally full of infusoria and entomostraca—exposed for many days to the abnormal temperature of this exceptional summer. This sample, therefore, is an enormously exaggerated instance of vegetable pollution, far in excess of anything that is likely to occur in connection with an ordinary water supply.

B is sewage as discharged into a brook, not allowed to stagnate, and not placed under circumstances exceptionally favourable to decomposition.

The permanent character of the vegetable as compared with the animal matter, and the sluggish development of ammonia in the case of the former, are here well illustrated.

If, then, it is necessary for the formation of nitrates that there should be a sufficient supply of ammonia, it is easy to understand why these salts are practically

derived from animal matter alone. It is not necessary for our present purpose to inquire into the reason of this difference between the two forms of organic matter, but the explanation is probably to be found not only in the smaller proportion of nitrogen, and the more permanent character of the nitrogenous constituents of vegetables, but also in the fact that these constituents are more effectually protected against the inroads of putrefactive organisms by the more resistant nature of the vegetable cell-wall.

Corroborative evidence is again afforded by numerous analyses of moorland waters, where the rain not only falls upon land covered with vegetation, but is often stored in bogs and marshes, in intimate contact with vegetable matters, and yet issues practically destitute of ammonia, nitrates, and nitrites.

Very instructive in this connection is a comparison of the results obtained by the Rivers Pollution Commissioners from the samples of water from gathering grounds out of reach of animal contamination with those afforded by waters from cultivated and inhabited districts: these results appear fully to justify the conclusion that "whilst the oxidation of animal matters in solution in water yields abundance of nitrates and nitrites, vegetable matters under like circumstances yield none or mere traces of these compounds."

We have yet to consider the second division of the evidence relating to the presence of nitrates in pure water, namely, examples of waters of known purity containing nitrogen in this form. And here we meet with the difficulty that no kind of proof is given, as a rule, of the freedom from contamination of those waters that are selected for the purpose of illustrating this point; and as other analytical data are seldom given at the same time, no opportunity is afforded to the critic of forming an opinion as to the character of the water.

Waiving, however, this point for the moment, if we examine the instances adduced by one of the most ardent opponents of nitrate determination, we find that the most pronounced specimen contains just 0.6 grain of nitrogen as nitric acid per gallon. Surely this affords no sufficient reason for ignoring the presence in well-water of ten, twenty, or even thirty times as much, implying a supply of nitrogen which, bearing in mind the quantity of water draining through a porous soil, assumes an almost commercial importance. In the face of the inevitable conclusion that the bulk of the nitrates contained in such waters can have no other origin than animal matter, it seems to me that their presence is a matter calling for most serious consideration.

Nor does the fact that fresh sewage contains no nitrates dispose of the advisability of their determination when present; for it is not, of course, suggested that the usual methods of detecting unoxidized nitrogen compounds should be neglected. It may, perhaps, be well to state clearly that I am not for a moment advocating the determination of nitrates to the exclusion of any other operation that can throw light upon the character of a water. I am merely endeavouring to show that this item is an absolutely essential feature of every water analysis.

It is now, I think, sufficiently apparent that, as stated previously, nitrates are all but exclusively derived from the oxidation of animal matters. Bearing, then, in

mind the danger besetting the ingestion of such matters, we have next to inquire how it can be maintained that water, containing nitrates obviously in excess of the maximum proportion that can be derived from the comparatively innocent sources reviewed in the preceding sections, can be held to be dietetically good. The only possible defence of this position would be a demonstration that during the conversion of the nitrogen of the animal matter into nitrates, the water necessarily undergoes such a process of purification as to remove the elements of danger which it is necessary to assume may accompany the original polluting substance. This brings us to the three remaining arguments against the determination of nitrates.

III. The suggestion that the process of oxidation involved in nitrification is in any true sense a destructive one appears to be a relic of the time when it was yet unsuspected that either the process itself or the communication of disease might be the work of living organisms, and could only be seriously advanced under a complete misapprehension of the process of nitrification, and a very unwarrantable confusion of the various forms of oxidation. Nitrification is itself essentially a physiological action on the part of living organisms, is completely arrested by a moderate rise of temperature, and is, in short, parallel in all respects with the formation of carbon dioxide in respiration. The process is carried out under conditions favouring microbial life in general, and especially conducive to the persistence in water of pathogenic forms for which moisture, an equable low temperature, and absence of light, are necessities.

IV. It is, then, further suggested that, nitrification being only carried out during percolation through soil, this latter process is a species of filtration during which all objectionable matters are removed from the water by mechanical retention, by chemical action, by the antagonism of other organisms, or simply by the failure of pathogenic organisms to persist in an unfavourable medium, all of which agencies may be supposed to have abundant opportunity of exercising their powers during the passage of polluted water through a considerable thickness of soil.

Now, there is a considerable appearance of reason about this argument; but to render it capable of practical application it is plainly necessary to define the minimum extent of such filtration necessary to effect purification, for we are now admittedly dealing with a water initially dangerously polluted; and then for the purposes of our present inquiry we have to ascertain how this minimum of filtration affects the question of nitrates.

Several empirical attempts have been made to meet this difficulty, and the first that naturally occurs to one is the distinction between deep and shallow wells. A deep well is defined as one the whole supply to which enters at a point not less than 100 feet from the surface, provided that the water really percolates through the soil, and is not conducted from the surface by fissures.

It was assumed by the Rivers Pollution Commissioners (*Sixth Report*, p. 89), on not very satisfactory grounds, that such a water, even if originally polluted, would be so far purified by filtration as to be a wholesome drinking water. It is, however, generally impossible to assure one's self of the fulfilment of the above conditions, and any evidence of unoxidized organic matter, or even of an exceptional amount of

oxidized nitrogen compounds, was held by the Commissioners to show that they were not perfectly carried out.

Then numerous investigators have suggested a limit of nitrogen (as nitrates) below which the evidence of pollution is too slight to be noteworthy; in other words, these chemists have suggested a chemical instead of a distance limit. This is generally placed at about 0.5 grain nitrogen per gallon. Thus we have seen that Mr. Ekin puts the maximum at .42 (0.6 part per 100,000); the Rivers Pollution Commissioners at 0.7 (1 part per 100,000), and so on. Lastly, we have the practical completion of nitrification, that is, the absence of significant quantities of unoxidized nitrogen, as the limit imposed by the holders of the views more immediately under discussion.

Now, all these proposals are no doubt the outcome of large experience in water analysis, but they cannot be said to be established upon thoroughly reliable premisses; and as the necessary information may be obtained from direct experiment where the conditions are absolutely under control, it appears to be very desirable to consider the question on this basis.

Before doing so, however, I wish to point out that the distance or "deep well" limit, and the quantitative limit of 0.5 grain nitrogen per gallon, are in practical accord, and having been obtained independently are all the greater corroboration one of the other.

Probably the fullest series of analyses of deep-well waters ever published occurs in the *Sixth Report of the Rivers Pollution Commissioners*, already alluded to; but unfortunately there are included amongst them several analyses not deserving that name. Still, of 157 samples not polluted with fresh sewage, fourteen only appreciably exceed the limit of 0.5 grain nitrogen as nitrates, and these are shown by the accompanying notes to be unsatisfactory in position, not sunk to the required depth, or otherwise not entirely in conformity with the definition. Some of them, indeed, have been closed on the ground of sewage contamination.

Perhaps the best possible illustration, however, is afforded by the deep wells in the chalk from which the Kent Water Company supplies part of the Metropolis; and this evidence is all the more valuable because it goes far to refute one of those misstatements which are copied with ever-increasing positiveness from one book to another until they are accepted as axioms! I refer to the alleged abnormal yield of nitrates by the chalk. One would be justified in interpreting the statements in many works to mean that there is hardly any limit to the proportion of nitrates a pure chalk water may exhibit. There is this much to be said in support of this assertion, that the chalk is probably of all formations the most suitable both in chemical composition and in physical structure for the active promotion of nitrification; and there is probably a greater yield of nitrates from a given amount of organic nitrogen, and a greater persistence of the nitrates so formed, than in any other formation. But it is no more true to say of the chalk that it furnishes nitrates *per se*, than it would be of any other formation. There must be abundant examples of chalk waters in the notebooks of most Southern analysts showing no abnormal proportion of nitrates; indeed, the average nitric nitrogen in sixty-six samples, including several obviously polluted

waters, was determined by the Rivers Pollution Commissioners to be 0.42 grain per gallon, or somewhat less than the average of samples from the New Red Sandstone.

However, to return to the Kent Water Company's works, these consist of several wells ranging from 150 to 500 feet in depth, the upper part of each well to a distance of 70 to 100 feet from the surface being lined with cast-iron cylinders to exclude superficial impurities. The maximum proportion of nitrates in this water is given by the Rivers Pollution Commissioners as 0.38 grain per gallon, the average being 0.287 grain. This agrees well with the average of 0.25, calculated from a series of monthly analyses extending over a period of ten years, and recorded by the late Dr. Tidy (*London Water Supply*, 1868-1878). Compare with this the proportion of 1.45 grain of nitric nitrogen yielded by the water of another well belonging to this company, but not, I believe, used for public distribution, this well being only 70 feet deep, and not protected against surface drainage.

Here, then, we have reasonable proof that water originally considerably contaminated with the products of decomposition of animal matters, after having undergone an amount of natural filtration considered under one system to guarantee the elimination of dangerous materials, has, if we adopt the chemical limit, lost the chemical evidence of pollution.

(To be continued.)

Methylene Blue as a Reagent for Sugar in Urine. N. Wender. (*Pharm. Post.*, 1893, xxvi. 393; through *Chem. Zeit.*)—One c.c. of the urine, previously diluted to 10 times its volume, is mixed with 1 c.c. of methylene-blue solution (1 gramme per litre) and 1 c.c. of normal potash solution in a test-tube; the mixture is diluted with 2 c.c. of water, and heated over a flame. The methylene blue will be completely decolorized if the original urine contained upwards of 0.5 per cent. of sugar. If the colour remain permanent the urine cannot be called diabetic. A. G. B.

The Amount of Fat in the Milk of Mecklenburg Herds. P. Vieth. (*Landwirthschaftliche Annalen d. Mecklenburgischen Patriotischen Vereins*, 1893, xvii., 131.)—From 1878 to 1885 Prof. Fleischmann determined in the laboratory of the Raden Dairy Institute the specific gravity, fat, and total solids of the milk of the Raden herd of cattle; samples of morning and evening milk were examined weekly. The results are:

			Variations.			Averages.
1878	2.84 to 3.93	3.40
1879	2.87 to 3.91	3.30
1880	2.92 to 3.82	3.27
1881	2.78 to 4.02	3.24
1882	2.89 to 3.58	3.20
1883	2.85 to 4.22	3.26
1884	3.04 to 3.95	3.30
1885	2.82 to 3.89	3.24

In the year 1886 the Government investigation was discontinued.

In 1885 the Rostock Dairy Company was started. For the first year the quantity of milk yielded by each herd only was noted, but in 1886 the fat was estimated by

Soxhlet's areometric method in weekly samples of morning and evening milk, and the monthly average calculated from these. The yearly averages are calculated from July 1 to June 30 in each year.

			Variations.			Yearly Averages.
1887-88	3.2 to 3.4	3.33
1888-89	3.1 to 3.7	3.43
1889-90	3.3 to 3.8	3.52
1890-91	3.3 to 3.6	3.46

The milk was supplied by 15 associates, and the variations in the composition of each is given in curves accompanying the paper. The average monthly composition calculated from the whole of them is—

		Fat.			Fat.
July	...	3.34%	January	...	3.40%
August	...	3.42	February	...	3.40
September	...	3.46	March	...	3.39
October	...	3.51	April	...	3.36
November	...	3.59	May	...	3.37
December	...	3.49	June	...	3.27

As the author has noticed in England, the amount of fat is highest in October and November and lowest in June. (See ANALYST, xvii. 84, and *Journ. Roy. Agric. Soc.*, xxv., s.s., Part I.)

H. D. R.

NOTE BY ABSTRACTOR.—It is hardly necessary to draw attention to the fact that the percentages given are obtained by Soxhlet's method, which gives about 0.25 per cent. less fat than Adams' process.

H. D. R.

Researches on the Adulteration of Olive-oil. V. Olivieri. (*Staz. Sper. Ag. Ital.*, xxiv. 387.)—The author has examined 106 samples of genuine olive-oil exhibited at the Palermo National Exhibition of 1891-92.

The amount of potash required for saponification varied from 19.05 per cent. to 19.50 per cent., and the iodine absorption from 79.0 per cent. to 83.2 per cent. The whole of the samples were examined in Amagat and Jean's oleorefractometer (see ANALYST, xv. 87); the variations found were from 0° to + 2°.

The following figures were yielded by the different oils examined by the author:

Name of Oil.	Deviation.	Potash Absorption.	Iodine Absorption.
Olive...	0° to + 2°	19.05 to 19.50	79.0 to 83.2
Cotton	+ 18.0°	19.1 to 19.55	104 to 108
Sesame	+ 15.5°	18.9 to 19.05	105 to 107
Colza	+ 26.5°	17.7 to 17.8	99 to 106
Earthnut	+ 7.5°	19.1 to 19.6	101 to 105
Poppy	+ 28.5°	19.2 to 19.5	133 to 138
"Olein"	+ 2.5° to + 4°	15.7 to 16.0	57.2 to 60.0

The olein examined was a bye-product of the manufacture of stearin candles, consisting of the liquid glycerides.

Mixtures were made as follows:

80% Olive	20% Cotton	gave a deviation of	...	+ 4.5°
60% "	40% Colza	"	...	+ 12°
70% "	30% Sesame	"	...	+ 4.5°
75% "	25% Earthnut	"	...	+ 2°

The author is of opinion that the oleorefractometer will be of use to detect considerable adulterations of olive oils; oils giving normal figures cannot always be passed as genuine without further examination. He relies on the iodine absorption to detect adulteration.

H. D. R.

NOTE BY ABSTRACTOR.—The maximum iodine absorption given by the author is low, as numerous cases of olive oils having iodine absorption of 87 per cent. to 88 per cent. have been recorded. Had he found such samples his conclusions would have doubtless been modified.

H. D. R.

Qualitative Examination of Phenacetin. C. Platt. (*Jour. Anal. and Applied Chem.*, 1893, vii. 77-83.)—The melting-point of pure phenacetin should be very close to 135° C., and if it fall to 133° C. the sample may be suspected as impure. The presence of acetanilide is the commonest cause of a fall in the melting-point, 5 per cent. of this impurity reducing the constant to 127° or 128° C. Inasmuch as the price of acetanilide is only one-fifteenth of that of phenacetin, it is sometimes added to increase the profit; but the objection to it does not end here, for Schroeder has shown that its decomposition in the system yields aniline. Antipyrin and parphenetidin should also be looked for in phenacetin.

The following tests will serve to identify phenacetin, and to prove its purity from the above adulterants:

1. Boil the sample with hydrochloric acid, dilute with water, cool, filter, and add two or three drops of potassium dichromate to the filtrate; if the sample be phenacetin, a ruby-red colour will be produced.

2. Boil with water, cool, filter, and add bromine water until the solution becomes yellow; pure phenacetin solution will remain clear.

3. Phenacetin will give a red colour when boiled with hydrochloric acid and ferric chloride.

4. Autenrieth and Hinsberg's test (*ANALYST*, 1892, 57).

5. Heat with alcohol and sulphuric acid; ethyl acetate will be evolved and recognised by its odour. At the same time aniline will be produced, and may be recognised by yielding phenylcarbamine when the solution is heated with chloroform and potash.

6. Alcohol will be evolved when phenacetin is boiled with potash; this distinguishes it from acetanilide.

7. To a well-cooled solution in strong hydrochloric acid add chlorine water: phenacetin gives a red colour; acetanilide a blue, which afterwards fades.

8. Potassium permanganate added to the hydrochloric acid solution gives a violet colour, which changes to dark ruby-red, with phenacetin, but a green colour with acetanilide.

9. Weak chromic acid gives a red colour in a dilute hydrochloric acid solution of phenacetin, but a yellow colour, becoming dark-green, with one of acetanilide; the dark-green solution gives a blue precipitate with potash.

10. The most delicate test for acetanilide in phenacetin is to heat with potash and chloroform, when acetanilide gives phenylcarbamine, but phenacetin does not.

11. The presence of antifebrin in phenacetin is detected by boiling the sample with a little water, cooling, filtering, and boiling the filtrate with a little nitrous acid and Plugge's reagent (a solution of mercurous nitrate with a little nitrous acid). Antifebrin will be betrayed by the production of a red colour.

12. To detect parphenetidin in phenacetin, melt 2.5 grammes of chloral hydrate in a test-tube, and add 0.5 gramme of the sample; pure phenacetin will dissolve to a colourless solution, becoming pink after some time. A trace of parphenetidin will produce a dark violet to a reddish or bluish violet, according to the quantity present.

A. G. B.

Saponification of Fats by Strong Sulphuric Acid. P. Schatzmann and H. Kreis. (*Chem. Zeit.*, 1893, xvii. 544, 545).—The authors point out that Pinette (ANALYST, 1893, p. 145) has failed to make their method a success because he has used sulphuric acid which is too strong. It was stated in the original paper on the subject (ANALYST, *loc. cit.*) that acid of sp. gr. 1.825 is used. The acid is now judged to be of the correct strength, not by the hydrometer, but by the way in which it dissolves pure butter and oleo-margarine when used in the way described in the original paper. Under these conditions pure butter should dissolve to a clear solution immediately, and oleo-margarine in 2 to 3 minutes, if the acid be of the correct strength. An acid which is too strong or too dilute will dissolve the fats too quickly or too slowly. Commercial oil of vitriol is generally too strong.

The acid which the authors use contains 91.53 per cent. H_2SO_4 , and forms an excellent qualitative test for margarine in butter fat. This acid gives good Reichert-Meissl numbers, and the authors insist that neither oxidation of sulphurous acid, nor elimination of it by a current of air or other gas (ANALYST, *loc. cit.*), is necessary.

That oxidation by permanganate before distillation gives too low a result is shown by the following figures:

		Reichert-Meissl Method.	Pinette Method.
Butter I.	...	29.1	18.8
„ II.	...	28.6	17.6
Margarine	...	7.1	5.3

A. G. B.

The Separation of Copper from Cadmium by the Iodide Method. P. E. Browning. (*Amer. J. Science*, 1893, xlv. 280-283).—The quantitative precipitation of copper as cuprous iodide is difficult on account of the perceptible solubility of the precipitate. Flajolot (*J. f. Prakt. Chem.* lxi. 105) recommends that the solution containing copper be brought to acidity with sulphuric acid, that a considerable excess of sulphurous acid be added, and that the precipitation be effected by hydriodic acid.

The author made a series of experiments, using about 0.12 grm. of copper for each, and precipitating under various conditions. He found a considerable deficiency of copper in the precipitate, save when the following precautions were adopted: The solution of the copper as sulphate, measuring about 25 c.c., is precipitated with 1.2 grms. of potassium iodide, the solution containing the precipitate being then evaporated to dryness to expel the free iodine. The residue is treated

with water, filtered on a Gooch filter, and well washed with hot or cold water. The asbestos constituting the filter should be fairly thick, and not allowed to run dry during the filtration, lest the filtrate tend to be turbid. The crucible and its contents are then dried in an air-bath at 120° – 150° C. to constant weight.

The possibility of precipitating copper quantitatively as cuprous iodide having thus been settled, it was found that the same method is applicable in the presence of varying proportions of cadmium as sulphate, and thus constituted a means of separation. The subsequent precipitation of the cadmium in the filtrate presents some difficulty, as when thrown down as sulphide in either acid or ammoniacal solution it filters slowly, a serious disadvantage, inasmuch as tedious washing to remove alkaline salts is inevitable. The author finally found that precipitation by means of sodium carbonate in a boiling solution—heating being continued until the precipitate became granular—was best under the conditions obtaining. The carbonate was collected on a Gooch filter, to avoid reduction on ignition, and heated until converted into oxide and weighed.

B. B.

Determination of Nitric Acid. C. F. Roberts. (*Amer. J. Science*, 1893, xlii. 126-134.)—The paper consists of a study of the conditions of accuracy of the Schloesing process for the estimation of nitrates, the following conclusions being arrived at:

(1) The nitric oxide should be passed through a solution of potassium iodide before collection, to decompose higher oxides of nitrogen.

(2) The nitric oxide may be collected over caustic soda, as proposed by Tie-mann, but it should not be left long in contact with this reagent, as it is perceptibly absorbed thereby.

B. B.

Rapid Method for the Detection of Iron in Commercial Copper Sulphate. G. Griggi. (*Boll. Chim. Farmac.*, 1893, xxxii. 549; through *Chem. Zeit.*)—5 c.c. of an aqueous solution (1:5) of the copper sulphate to be tested are poured into a test-tube, and 5 c.c. of an ethereal solution of salicylic acid (1:10) gently added. Should the copper sulphate be free from iron, the junction of the two solutions remains colourless, but in the presence of iron a violet colour appears, varying in intensity according to the amount of iron present.

B. B.

The Content of Lecithin in Butter. E. Wrampelmeyer. (*Landw. Versuchs.*, 1893, xlii. 437; through *Chem. Zeit.*)—The hope of discriminating between butter and margarine by means of their respective contents of lecithin cannot be realized, for although a difference exists, it is by no means constant. Thus, the author found an amount of phosphoric acid in the ash of butter corresponding to 0.045% of the original butter. Margarine similarly treated gave 0.016%. These results were obtained on the unfiltered fats, and therefore included the phosphoric acid in the salts accompanying the fat itself. Experiments on the filtered fats showed that at most a trace of phosphoric acid occurred in margarine, while butter fat, although containing more than a trace, was poorer in this constituent than the butter from which it was derived. Moreover, the percentage fluctuated, and thus no useful standard could

be fixed. The quantity of lecithin calculated from the amount of phosphoric acid in the filtered fat averaged 0.017%, in place of tenfold that amount hitherto accepted as being present.

B. B.

"SUBSTANCE, NATURE, AND QUALITY."

IMPORTANT LEGAL DECISION.

HIGH COURT OF JUSTICE.—QUEEN'S BENCH DIVISION.

BEFORE MR. JUSTICE CHARLES AND MR. JUSTICE WRIGHT.

BAKEWELL, *Appellant*—DAVIS, *Respondent*.

(*From the Times, November 7, 1893.*)

THIS case yesterday raised a question novel and important under the Adulteration Acts—that a vendor of milk which is undoubtedly poor, but which is not shown to have been made so by any admixture of water, etc., and can only be inferred from the very pooriness to be so from withdrawal of cream, can be convicted under the Adulteration Acts. It was an appeal against a conviction under the Adulteration Acts of a farmer for selling milk "containing 22 per cent. of fat less than natural." It was proved that the prosecutor, an inspector, procured a sample of milk from a churn of milk in course of delivery at a railway-station in Birmingham from Bakewell to the North Staffordshire Dairy Company, in pursuance of a contract. The churn had a label on it, "The contents of this churn are warranted to be new and pure milk." The public analyst, in whose presence the material operations took place, proved that there were in the milk 88 parts and a fraction of water, and that the total solids were 11, 35 per cent. of which solids, not fat, were 8.66, and solids, fat, 2.69, and he stated that the sale of any milk in which the percentage of fat was less than 3 per cent., was fraudulent, but he admitted that from the dryness of the season (this was in April last), poor feeding, or other causes, the fat in the milk from one cow might fall below that standard, and he had known a cow yield only 2.6 per cent. of fat. But he was of opinion that there had been no actual adulteration by mixture of water. The appellant gave evidence that the milk was from a dairy of twenty-six cows, and that he had taken no cream from it, and ascribed the pooriness of the milk to the dryness of the season or the quality of the food, and other evidence was given to show that the pooriness of the milk arose from natural causes. It was contended that, as there was no evidence of actual adulteration, and the pooriness of the milk might be from natural causes, the defendant could not be convicted. The magistrates, however, were of opinion that the appellant, though he did not know of anything wrong in the milk, did not prove that the milk was genuine or that the absence of fat arose from natural causes. They accordingly found that the milk was not of the nature, substance and quality of the article demanded, and that it had been sold by the appellant under a contract of sale to the prejudice of the purchaser, and they convicted him accordingly, but they stated a case to raise the question whether their conviction was correct in point of law.

Mr. Poland, Q.C. (with Mr. Wills), appeared for the appellant in support of his appeal, and urged that the magistrates had not found as a fact that any cream or fat had been abstracted from the milk, and selling poor milk was, he said, no offence, unless it was so by adulteration or withdrawal.

Mr. Jelf, Q.C. (with Mr. William Graham and Mr. Hugo Young), appeared for the prosecutor in support of the conviction, and urged that the certificate of the analyst was sufficient, and showed ample grounds for conviction in stating that the milk was not pure and genuine.

Mr. Wills, in reply, urged that there was no finding that the milk was poor by reason of withdrawal of cream. Neither abstraction of cream nor addition of water was charged or

found, and no offence under the Act was charged, for selling poor milk *per se* was not an offence. [Mr. Justice Charles : There are cases in which a seller may be liable without guilty knowledge.—Mr. Justice Wright : Suppose the man half starves his cow, and so the milk is poor. Has he a right to sell milk not genuine, nor of the nature or quality of good milk ?] The conviction is under section 9, which requires a bad intent. [Mr. Justice Charles : No ; under section 6, "not of the substance, nature, or quality of the article demanded." It follows the words of that section.] The analyst's certificate really, though indirectly, charges abstraction, as it speaks of abstraction as a fraud, and of that there was no evidence, and so it is bad, for by the Act the certificate is made evidence. [Mr. Justice Charles : Only of "the facts stated." It may be that the observations ought not to have been inserted.—Mr. Justice Wright : There was no evidence of any moral fraud or any intentional injury.] No evidence of abstraction of cream, and yet it is indirectly charged in the certificate.

Mr. Justice Charles, in giving judgment, said the defendant was charged, it was quite clear, under section 6, as to selling something not of the substance, nature, or quality of the article demanded. The analyst's certificate was to the effect that the milk contained much less cream than natural and usual, and less than a certain proportion, he added, would be fraudulent. That observation was unauthorized, but it was urged that it rendered the certificate invalid altogether, as it conveyed a charge of wilful abstraction. The magistrates, however, had expressly acquitted the defendant of that, and he did not think the analyst's statement was a statement of fact. It was a mere statement of opinion, which the magistrates did not adopt or act upon. He did not think, therefore, that the certificate was altogether bad or void. The main question was whether it was sufficient, and it in effect stated the results of the analysis. The result of the analyst's certificate was that the proportion of cream or fat in the milk was less than natural or usual. That was the proper course, and it showed that an offence against the statute had been committed.

Mr. Justice Wright concurred, observing that, as to the analyst's certificate, it was only in cases of adulteration that it was necessary to enter into the proportion of ingredients as showing fraud.

Appeal accordingly dismissed, and conviction upheld.

REVIEWS.

REACTIONS: A SELECTION OF ORGANIC CHEMICAL PREPARATIONS IMPORTANT TO PHARMACY IN REGARD TO THEIR BEHAVIOUR TO COMMONLY-USED REAGENTS. By F. A. FLÜCKIGER, Ph.D., M.D. Translated, revised, and enlarged by J. B. NAGELVOORT. Authorized English edition. Detroit, Mich., U.S.A.: George S. Davis. 1893. Price 2 dollars.

This book is one which, from the high repute of the author, will receive a welcome at the hands of most practising analysts. It contains succinct descriptions of the properties of and tests for a variety of organic preparations used in pharmacy. In the translator's preface it is stated that "Very elaborate and full information on many articles treated of in this small volume, in a modest way, appear in the recently published second part of vol. iii. of Allen's 'Comm. Org. Analysis,' and in Guareschi's 'Introduzione allo Studio degli Alcaloidi,' Torino, 1892. Where the statements of Allen and Flückiger or other authorities are not concordant, I have given the results of personal experience. This has given me an opportunity to decide some few moot problems in analytical work." The spelling adopted for

scientific and chemical words is that laid down by the American Association for the Advancement of Science, 1891, and it seems a great pity that this barbarous plan has received the sanction of the translator. It is difficult to perceive the advantage of spelling "chloride" without the final "e," of writing "sulfur" for sulphur, and still more to be deprecated is the system of dropping the final "e" for bodies of basic nature, such as morphine, strychnine and berberine.

Some of the translation of scientific terms from the German cannot be regarded as happy, or even accurate. For instance, the rendering (on page 118) of "sulphobenzaminsauresammoniak" as "sulphobenzoic ammonia;" and of "ferri-sulphobenzaminat" as "sulpho-benzoate of iron." Again, on the following page, "nitro-prussid-sodium" is neither English nor elegant.

The work is admirably printed, and both the empirical and structural formulæ of most preparations are given. The tests are described tersely, but with amply sufficient working detail, and the conditions of success are clearly laid down.

Altogether, the work is to be recommended as a most useful book of reference, containing in a compact form a large amount of information on articles the examination of which is troublesome and definite information widely distributed or difficult to obtain.

A. H. A.

A GUIDE TO THE EXAMINATION OF THE URINE. By J. WICKHAM LEGG, F.R.C.P.

Seventh edition. Edited and revised by H. LEWIS JONES, M.A., M.D.,
M.R.C.P. London: H. K. Lewis. Price 3s. 6d.

This well-known handbook, which has now reached its seventh edition, has for many years enjoyed a well-deserved popularity amongst medical practitioners and students. The present edition has been carefully revised, and in part rewritten, by Dr. Jones. Without pretending to be an exhaustive treatise on urine, it contains sufficient information for the ordinary requirements of the clinical clerk or the busy practitioner. The various methods described, though given briefly and concisely, contain all the necessary details for their successful execution. The information given is thoroughly accurate, reliable, and well up to date. In the chapter on the use of the polariscope for the determination of glucose or albumin in the urine, it is difficult to discover what form of instrument is referred to. Any instrument with a scale for cane-sugar would obviously give erroneous indications for glucose or albumin. The only instrument we are acquainted with which gives a direct reading in percentages of glucose or albumin is the Mitscherlich half-shadow, made by Schmidt and Hänsch. To attain this object a set of tubes of special length are supplied with this cheap and handy little instrument for the use of medical practitioners. The book is neatly printed, well illustrated, and of a handy size. We can cordially recommend it as a reliable "Guide."

W. J. S.

APPOINTMENT.

Mr. W. W. Fisher, M.A. (Oxon.), F.I.C., Public Analyst to the County of Oxford, has been appointed by the County Council as the District Agricultural Analyst under the Fertilizers and Feeding Stuffs Act, which comes into force on January 1, 1894.

The Analyst,

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INDEX TO SUBJECTS.

A.

	PAGE
Acetic acid and pyroligneous acid synonymous trade terms	28
Acetic acid, Note on the Valenta test, CHATTAWAY, PEARMAN & MOOR ...	147
Acetic acid, turbidity Temperatures of oils and fats with, E. W. T. JONES ...	151
Acetic acid turbidity test, Results of, on various oils and fats	150, 152
Acetic acid, Discussion on the Valenta test for oils and fats	152
Acetanilide and alkaloids, The resemblance in the reactions of, E. SCHÄR ...	279
Acetone in alcohol, Methods for the detection of	267, 268
Acid, Determination of free, in solutions of the salts of the heavy metals, F. HOFFMANN	70
Acid, Influence of strength of, in the Leffmann-Beam process	62
Adulteration, Two disputed cases of, E. G. Clayton	220
Adulteration, The need for fuller statistics of, C. H. CRIBB	273
Albuminoids, The decomposition of, by alkaline hydrates, V. VERDRÜDI ...	251
Alcohol in vinegar	13
Alkaloids in drugs, The volumetric determination of, CASPARI & DOHME ...	24
Alkaloids and acetanilide, The resemblance of the reactions of, E. SCHÄR ...	279
Alkali in alkaline arsenites, The determination of, G. FAVREL	42
Alum, aluminium hydroxide, and aluminium phosphate, Influence of, on the digestibility of bread, BIGELOW & HAMILTON	253
Analysis, Some frequently neglected errors in, H. D. RICHMOND	99
Annual meeting	25
Annual address, President's	49
Annual dinner	57
Arsenic, Products of the sublimation of, J. W. RETTGERS	47
Arsenites, Alkaline, The determination of alkali in, G. FAVREL	42
Antimony and tin, Separation of, in alloys, MENGIN	254
Apparatus for the extraction for analysis of gases dissolved in water, S. HARVEY ...	121
Atropine and strychnine, Distinctions between by Vitali's reaction, VITALI ...	255

B.

	PAGE
Baking-powder not an article of food (legal decision)	48
Balances, Errors due to differences in load of	100
Beef-fat in lard, Estimation of, W. F. K. STOCK	2
Bread, Influence of aluminium salts on the digestion of, BIGELOW & HAMILTON ...	253
Bromine in urine, The determination of, A. NICOLLER	23
Butter, The proximate composition of, H. D. RICHMOND	16
„ Average amount of water in	16, 17
„ The cryoscopic method in the examination of, GARELLI & CACCANO	70
„ Average composition of, during 1893	75
„ Analyses of old samples	76
„ On the change in the composition of, by long keeping, ALLEN & MOOR	128
„ analysis, Recent modifications of the Reichert-Meißel method of, C. BUNTR	188
Butter-fat, Determination of volatile and insoluble fatty acids in, W. H. BEAL ...	252

C.

Calcium, Amount of carbon dioxide combined with, in water	124
Carbonic anhydride in the presence of soluble sulphides, Determination of, A. WOLKOWICZ	189
Cheese, Cheshire, C. M. BLADES	131
„ Remarks on estimation of fat in ...	132
„ Remarks on proteids of	132
„ On the composition of, CHATTAWAY, PEARMAN & MOOR	145
„ Results of the analyses of various ...	146
„ Estimation of fat in, S. BONZYN-SKI	250
Chromium in chromium-steel, Determination of, SPÜLLER & KALMAN	116
Churchyards, Analyses of waters from wells in close proximity to, J. A. VOELCKER	107
Chlorine in saltpetre, The occurrence of, A. HELLIÖH	221
Chlorine in the presence of bromine and iodine, Detection of, NIELLIERS & FAYOLLE ...	258

	PAGE		PAGE
Cider vinegar, G. S. COX	89	Fats and oils, The methods of testing, E. MILLIAU	118
Cider vinegar, Note on Mr. Cox's paper on, A. H. ALLEN	91	Fats and oils, Turbidity temperatures of, with acetic acid, E. W. T. JONES	151
Cloves, Estimation of essential oil in, W. LENZ	250	Fats and resins, A new method of analysing, P. C. McILHINEY	141
Cocoa, The quantitative separation and estimation of the alkaloids of, W. E. KUNZE	194	Fatty acids, Method for determining the freezing-point of, F. WOLFBAUER	251
Copper, The determination of impurities in commercial, B. BLOUNT	92	Freezing-points of fatty acids, Method of determining, F. WOLFBAUER	251
Copper, The determination of foreign metals in, W. HAMPE	93	Furfural as a reagent for sesame-oil in other oils, VILLAVECCHIA & FABRIS	47
Copper, lead, tin, and iron, Volumetric estimation of minute quantities of, BUDDEN & HARDY	169	Fusel-oil, The influence of, in the Leffmann-Beam process	62
CORRESPONDENCE:—		G.	
Between Mr. Allen and Mr. Bannister	231	Gases in water analysis, Simple apparatus for the extraction of, S. HARVEY	121
Letter from Mr. Edward Bevan	216	Ginger, On the detection of exhausted, ALLEN & MOOR	124
Letter from Mr. Edward Collens	96	„ On extraneous mineral matter in commercial, A. H. ALLEN	217
Letter from Mr. A. F. de Hailes	240	„ Proportion of ash yielded by 104 samples of genuine and commercial	218
Letter from Messrs. Richmond and Bosseley	96	„ Opinion of Somerset House Authorities on sample of	219
Letter from Mr. W. J. K. Stock	264	„ Yield of ash in 58 samples of ground	219
Cotton-seed-stearin and maize-oil, F. HART	42	Glass, Comparative tests of different sorts of, with reference to their chemical composition, F. FOERSTER	282
Cotton-seed-oil in lard, Note on the detection of, E. J. BEVAN	83	H.	
Cream, Observation on the analysis of	84	Hops, Estimation of the tannin-bodies in, M. HAYDUCK	139
D.		Horse-flesh, The chemical recognition of, BRAUTIGAM & EDELMANN	24
Discussion on the vinegar question	8, 26	I.	
Draft of a Sale of Food and Drugs Act prepared by the Council of the Society of Public Analysts	156	Indiarubber goods, The analysis of, D. HOLDE	43
E.		„ The analysis of, LOBRY DE BRUYN & VAN LEEUT	109
Errors in analysis, Some frequently neglected, H. D. RICHMOND	99	„ The analysis of, R. HENRIQUES	111
Ether, A simple method of purifying commercial, M. EKENBERG	280	Indicators for volumetric analysis	255
Ethereal oils, The analysis of, J. KLIMONT	222	Iron, lead, copper, and tin, Colorimetric estimation of minute quantities, BUDDEN & HARDY	169
Extracts from the evidence given before the Select Committee on Food Products Adulteration of 1894:—		Iron, Determination of silicon and aluminium in, L. L. DE KONINCK	207
1. Evidence referring to the Work done at Somerset House, and the Relation between Public Analysts and the Referees	208	K.	
2. Evidence relating to milk	212	Kapok-oil, R. HENRIQUES	44
3. „ milk standards	225	Kjeldahl-Willfarth and Stock methods of determining nitrogen, Comparison of, CAVAZZANI & CECCONI	224
4. „ decomposed milk	228	L.	
5. „ margarine and butter	259	Lactic acid, Volatility of	244
6. „ the amount of water in butter	263	Lard, Note on the detection of cotton-seed oil in, E. J. BEVAN	88
7. „ vinegar	283		
8. „ ginger	284		
9. „ lard	284		
10. „ sugar	284		
11. „ standards and limits	284		
F.			
Fat in cheese, Estimation of, S. BONDZYN-SKI	250		

INDEX.

	PAGE
Lard, Qualitative tests for purity of, SAMUELSON ...	251
„ Contribution to the analysis of, G. HALPEN ...	282
„ Estimation of beef-fat in, W. F. K. STOCK ...	2
Lead, Separation and volumetric estimation of, LINDEMANN & MOTTEAU ...	94
Lead, copper, tin, and iron, Colorimetric estimation of minute quantities of, BUDDEN & HARDY ...	169
Leffmann-Beam method of fat estimation in milk, RICHMOND & BOSELEY ...	62
Leffmann-Beam method, Suggestions for the practical use of ...	66
Legal decision, "Baking-powder not an article of food" ...	48
M.	
Mace, Estimation of essential oil of, W. LENZ ...	250
Maize-oil and cotton-seed stearin, F. HART ...	42
Margarine, The addition of phenolphthalein to, F. HART ...	69
Metals, Pyrological method of estimating minute quantities of ...	175
Metals in aerated beverages, Hehner's method of determining ...	177
Methylated spirit in tinctures, spirits, etc., Detection of, A. ASHBY ...	265
Methyl alcohol in ethyl alcohol, Processes for the detection of by:—MM. RICHE & BARDY, J. T. MILLER ...	266
A. DUPRÉ, O. HEHNER, J. E. REYNOLDS ...	267
P. CAZENÈVE, M. DUCLAUX ...	268
Milk, Average composition of, during 1893 ...	74
„ Analysis of separator slime ...	86
„ and milk products, Composition of, H. D. RICHMOND ...	73
„ Determination of the specific gravity of curdlet, M. WEIBULL ...	24
„ The Leffmann-Beam method of fat estimation in, RICHMOND & BOSELEY ...	62
„ Observations on the ash of ...	83
„ Presence of nitrates in ...	83
„ skim-milk, and whey, C. B. COCHRAN ...	119
„ Difference between the casein of human and cow's ...	206
„ The loss of total solids on keeping, E. J. BEVAN ...	241
Milk-scale, An improvement in, RICHMOND's, CASSAL & GERHANS ...	37
„ An adjunct to, H. D. RICHMOND ...	38
Milk analysis, addition of preservatives to milk intended for analysis ...	247
„ „ Formaldehyde as a preservative for milk in ...	247
„ „ New formula for calculation in ...	81
„ „ Comparison of the Adams and Werner-Schmid processes of ...	78

	PAGE
Milk analysis, Examination of crysotile method of ...	77
„ „ Suggestions as to duplicates being forwarded to referees at once ...	250
„ „ Precautions to be observed in the Adams method of ...	78
„ „ Worthlessness of time allowance in ...	246

N.

Naphtha for methylating, Proposed standard for ...	271
--	-----

O.

Oils, essential, especially in cloves and mace, Estimation of, W. LENZ ...	250
Oils, ethereal (essential), The analysis of, J. KLIMONT ...	222
Oils and fats, Methods of testing, E. MILLIAU ...	118
Oils and fats, Turbidity temperature of, with acetic acid, E. W. T. JONES ...	151
OBITUARY NOTICES:— Dr. Arthur Hill Hassall ...	97
Dr. C. R. Alder Wright ...	193

P.

Parliamentary Committee on Food Products Adulteration of 1894, Extracts from evidence of ...	208, 225, 259, 283
Peas, The occurrence of poisonous leguminous seeds in Indian, J. A. VOELCKER ...	102
Peel, Note on lemon and orange, E. G. CLAYTON ...	134
Pepper, Composition of, E. VON RAUMER ...	45
Perchlorates, Detection of, in the presence of chlorides, chlorates & nitrates, GOOCH & KREIDLER ...	224
Phosphor tin, Note on the analysis of, F. L. TEED ...	133
Phosphoric acid, The determination of by the titration of the yellow precipitate with standard alkali ...	95
Phosphoric acid, On the determination of, H. PEMBERTON ...	135
Phosphates, Untreated mineral and basic slag, Estimation of citric-soluble phosphoric acid in, P. WAGNER ...	205
Platinum, Permeability of at a red heat to reducing gases ...	101
Poisonous leguminous seeds in Indian Peas, The occurrence of, J. A. VOELCKER ...	102
Proximate composition of butter, The, H. D. RICHMOND ...	16
Pyroligneous acid and acetic acid synonymous trade terms ...	28

R.

Reichert-Meißl method, Recent modifications of, C. BUNTE ...	188
Resins and fats, A new method of analysing, P. C. McILHINEY ...	141

	PAGE		PAGE
REVIEWS:—		Sulphuric acid, Table for deducing strength	
Adulteration [Agricultural fertilizers & feeding stuffs], F. A. CRIPPS-DAY ...	287	from specific gravity ...	68
Animal and vegetable fixed oils, butter, waxes: their preparation, properties and uses, C. R. ALDER WRIGHT ...	143	Errors in standardizing ...	101
Fertilizers and feeding stuffs, BERNARD DYER, D.Sc. ...	264	The volumetric estimation of, W. WINDISCH ...	138
Handbuch der Stickstoffhaltigen Orthocondensations Producte, O. KÜHLING	120		
Johnston's elements of agricultural chemistry, C. M. AIKMAN ...	72	Tannin-bodies of hops and their estimation,	
Micro-organisms of fermentation, The, ALFRED JÖRGENSEN ...	95	M. HAYDUCK ...	139
Micro-organisms in water, Dr. and Mrs. PERCY FRANKLAND ...	192	Tin, Note on the analysis of phosphor-, F. L. TRED ...	133
Manures and manuring, C. M. AIKMAN	71	Tin, lead, copper and iron, Colorimetric estimation of minute quantities of, BUDDEN & HARDY ...	169
Nature's Hygiene, C. T. KINGZETT ...	191	Tin and antimony, Separation of, in alloys, MENGIN ...	254
Principles and practices of agricultural analysis, H. W. WILEY ...	72	Tinctures, spirits, etc., The detection of methylated spirit in, A. ASHBY ...	265
S.		U.	
Saccharin in wine, liqueurs, or beer, E. SPARTH ...	42	Urine, Determination of bromine in, A. NICOLLE ...	23
Sale of Food and Drugs Act, Draft for, prepared by the Council of the Society of Public Analysts ...	156	Disturbing influence of glycuronic acid in sugar estimation in, ...	184
Saltpetre, The occurrence of chlorine in, A. HELLMICH ...	221	Examination of, for small quantities of sugar, A. H. ALLEN ...	178
Separation and estimation of antimony, tin and arsenic by sodium peroxide ...	281	Method of separating creatinine from ...	182
Sesame-oil in other oils, Furfural as a reagent for, VILLAVECCIA & FABRIS ...	47	New method of using Pavy's solution in testing ...	181
Slag, basic, and untreated mineral phosphates, Estimation of citrate-soluble phosphoric acid in, P. WAGNER ...	205	Occurrence of sugar in normal, views for and against ...	178
Sodium peroxide in analysis, Utilization of, O. KASSNER ...	280	Phenylhydrazine as a test for sugar in	183
SOMERSET HOUSE ANALYSES:		Substances interfering with Fehling's test for sugar in ...	180
Milk ...	220		
Ginger ...	219	V.	
Chicory and coffee ...	221	Valenta test for acetic acid, Note on, CHATT- AWAY, PEARMAIN & MOOR ...	147
Statistics of adulteration, The need for fuller, C. H. CRIBB ...	273	Vinegar, E. COLLENS ...	33
Steel, Determination of chromium in chromium ...	116	Cider, G. S. COX ...	89
Stock and Kjeldahl-Willfarth methods of determining nitrogen, Comparison of, CAVAZZANI & CRECONI ...	224	Note on Mr. Cox's paper on cider-vinegar, A. H. ALLEN ...	91
Sugar in urine, Examination for minute traces of, A. H. ALLEN ...	178	Definitions of ...	9, 10, 11
Sugars, Colour-reaction for the detection of, FISCHER & JENNINGS ...	224	Typical analyses of ...	15
Sulphides, Determination of carbonic anhydride in the presence of soluble, WOLKOWICZ ...	189	Legal decision as to the strength of	26
Variation in colour due to formation of hydrosulphides ...	176	Trade definitions of the strength of	27
Effect of citric or tartaric acid on metallic ...	176	Volumetric analysis, Ammonium chloride as a standard in ...	257
Sulphide of lead, Effect of sugar on colour of precipitate ...	176	Contributions to, B. REINITZER ...	255
Sulphuric acid, Methods of estimating strength of, ...	63	Influence of boiling in glass vessels in ...	257
		Method for causing precipitates to subside quickly in, P. N. RAIKOW ...	143
		Preparation of pure sodium carbonate for	256
		Volumetric apparatus, Errors due to ...	100

	PAGE
Volumetric determination of alkaloids in drugs, CASPARI & DOHME ...	24
Volumetric estimation of sulphuric acid, W. WINDISCH ...	138
W.	
Water analysis, The interpretation of results, F. W. STODDART...	18, 39

	PAGE
Water analysis, Simple apparatus for the extraction of gases dissolved in water, S. HARVEY	121
Waters from wells near churchyards, Analysis of, J. A. VOELCKER ...	107
Weights, Errors due to incorrect ...	99
Whey, milk and skim milk, C. B. COCHRAN	119

INDEX OF AUTHORS' NAMES.

A.	
	PAGE
ALLEN, ALFRED H., Note on Mr. Cox's paper on cider-vinegar ...	91
„ The examination of urine for small quantities of sugar	178
„ On the extraneous mineral matter contained in commercial ginger ...	217
ALLEN & MOOR, On the detection of exhausted ginger ...	124
„ On the change in the composition of butter on long keeping ...	128
ASHBY, ALFRED, On the detection of methylated spirit in tinctures, spirits, etc. ...	265
B.	
BRAL, W. H., Determination of volatile and insoluble fatty acids in butter-fat	252
BEVAN, EDWARD J., The loss in total solids of milk on keeping...	241
„ Note on the detection of cotton-seed-oil in lard ...	88
BIGELOW & HAMILTON, The influence of alum, aluminium hydroxide, and aluminium phosphate on the digestibility of bread ...	253
BLADES, CHARLES M., Cheshire cheese	131
BLOUNT, BRETRAM, The determination of impurities in commercial copper	92
BONDZYNSKI, F., Estimation of fat in cheese ...	250
BRAUTIGAM & EDELMANN, The chemical recognition of horseflesh ...	24
BRUYN L. DE & VAN LEEUW, The analysis of rubber goods ...	109
BUDDEN & HARDY, Preliminary notes on the colorimetric estimation of minute quantities of lead, tin, copper and iron ...	169
BUNTE, C., The recent modifications of the Reichert-Meissl method ...	188

C.	
	PAGE
CASSAL & GERRANS, An improvement in Richmond's milk-scale ...	37
CASPARI & DOHME, The volumetric determination of alkaloids in drugs ...	24
CAVAZZANI & CRECONI, Comparison of the Kjeldahl-Willfarth and Stock methods of determining nitrogen ...	224
CHATTAWAY, PEARMAIN & MOOR, On the composition of cheese ...	145
CHATTAWAY, PEARMAIN & MOOR, On the Valenta acetic acid test ...	147
CLAYTON, E. G., Two disputed cases of adulteration...	220
„ Note on lemon and orange peel ...	134
COCHRAN, C. B., Milk, skim-milk and whey	119
COLLENS, EDWARD, Vinegar ...	33
COX, G. S., Cider vinegar ...	89
CRIBB, CRECIL H., The need for fuller statistics of adulteration ...	273
E.	
EKENBERG, M. A., Simple method of purifying commercial ether ...	280
F.	
FAVEEL, G., The determination of alkali in alkaline arsenites ...	42
FISCHER & JENNINGS, Colour reaction for the detection of sugars ...	224
FORRESTER, F., The comparative tests of different sorts of glass with reference to their chemical composition ...	282
G.	
GARELLI & CARCANO, The use of the cryoscopic method for the examination of butter ...	70
GOOCH & KREIDLER, Detection of perchlorates in the presence of chlorides, chlorates and nitrates ...	224
H.	
HALPEN, G., Contributions to the analysis of lard ...	282
HAMPE, W., Determination of foreign metals in commercial copper ...	93

	PAGE		PAGE
HART, F., The addition of phenolphthalein to margarine	69	REINITZER, B., Contributions to volumetric analysis	255
„ Cotton-seed stearin in maize-oil	42	RETTGERS, W. J., The products of the sublimation of arsenic	47
HARVEY, SIDNEY, A simple apparatus for the extraction for analysis of the gases dissolved in water... ..	121	RICHMOND, H. DROOP, Some frequently-neglected errors in analysis	99
HAYDUCK, M., The tannin-bodies of hops, and their estimation	139	„ The composition of milk and milk-products	73
HELLICH, A., The occurrence of chlorine in saltpetre	221	„ An adjunct to the milk-scale	38
HENRIQUES, R., The analysis of rubber goods	111	„ The proximate composition of butter	16
„ Kapok-oil	44	RICHMOND & BOSELEY, The Leffmann-Beam method of fat-estimation in milk	62
HOFFMANN, F., Determination of free acids in solutions of the salts of the heavy metals	70		
HOLDE, D., The analysis of rubber goods... ..	43	S.	
J.		SAMUELSON, DR., On lard	251
JONES, E. W. T., Turbidity temperatures of oils and fats with glacial acetic acid	151	SCHÄR, E., Resemblance between the reactions of the alkaloids and acetanilide... ..	279
K.		SPÄTH, E., The detection of saccharin in wines, liqueurs and beer	42
KASNER, O., Utilization of sodium peroxide in analysis	280	SPÜLLER & KALMAN, Determination of chromium in chromium-steel	116
KLIMONT, J., The analysis of ethereal oils	222	STOCK, W. F. KEATING, On the estimation of beef-fat in lard	2
KONINCK, L. L. DE, Determination of silicon and aluminium in iron	207	STODDART, F. W., Water analysis: the interpretation of results	18, 39
KUNZE, W. E., The quantitative separation and estimation of the alkaloids of cocoa... ..	194	T.	
L.		TRED, F. L., Note on the analysis of phosphor-tin	133
LEHMANN & HEMPEL, Milk, distinction between human and cow's	206	V.	
LENZ, W., Estimation of essential oils, especially in cloves and mace	250	VEEDRÖDI, V., The decomposition of albuminoids by alkaline hydrates	251
LINDEMANN & MOTTEAU, The separation and volumetric estimation of lead	94	VILLAVECCHIA & FABRIS, Furfural as a reagent for sesame-oil in other oils	47
M.		VITALI, D., Distinctions between atropine and strychnine by Vitali's reaction	255
MCLHINEY, P. C., New method of analysing fats and resins	141	VOELCKER, J. A., Analysis of waters from wells in close proximity to churchyards	107
MENGIN, Separation of tin and aluminium in alloys	254	„ The occurrence of a poisonous leguminous seed in Indian peas	102
MILLIAU, The methods of testing fats and oils	118	W.	
N.		WAGNER, P., Estimation of citrate-soluble phosphoric acid in basic slag and in untreated mineral phosphates	205
NICOLLE, A., Determination of bromine in urine	23	WEISULL, M., Determination of the specific gravity of curdled milk	24
NIELIERS (VILLIERS) & FAYOLLE, The detection of chlorine in the presence of bromine and iodine	258	WINDSCH, W., The volumetric estimation of sulphuric acid	138
P.		WOLFBauer, F., Method for the determination of the freezing-point of fatty acids	251
PEMBERTON, H., JUN., On the determination of phosphoric acid	135	WOLKOWICZ, A., Determination of carbonic anhydride in the presence of soluble sulphides	189
R.			
RAIKOW, P. N., An improved volumetric precipitation process	143		
RAUMER, E. VON, Composition of pepper	45		

THE ANALYST.

JANUARY, 1894.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on December 6th at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner occupied the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed as members : Dr. J. Duncan, St. Petersburg ; Percy Andrews Ellis Richards, A.I.C., Charing Cross Hospital, W.C. ; and Robert Walter Oddy, F.I.C., Laboratory, Walterhouse, Toad Lane, Rochdale.

The following gentlemen were duly elected members : J. Kear Colwell, Great Russell Street, W.C. ; and H. H. B. Shepherd, Upper Clapton, N.E.

The Chairman (Mr. Hehner), referring to the death of Mr. R. H. Davies, late Secretary to the Society, said : As a Society, and as members of the profession of analytical chemists, we have suffered a great many losses, but on few occasions have we been so deeply touched as on the present one, when, as you know, we have to lament the death of one of our Secretaries, Mr. R. H. Davies. All of us had been aware that he had been ill, hopelessly ill, for many months past, and the most sanguine amongst us could not have expected that he would have been spared very long. He died at Bournemouth on November 16th. Mr. Davies was not a very old member of the Society ; he has been a particularly active and prominent member, both in his private capacity and as secretary. All of us who came into intimate contact with him can have nothing but pleasant and affectionate recollections of him. His opinions were decided and sound, he was possessed of a remarkably clear intellect, and as a public analyst he was as popular as a public analyst could be. By those of us who met him as a friend he will never be forgotten. The Council, by virtue of one of our bye-laws, have to-night filled up the vacancy created by the death of Mr. Davies, and have unanimously recommended Mr. Bevan, who I need not introduce to you, to fill the vacant post. He has kindly consented to act until the ballot-papers electing the new Council are opened at the Annual Meeting in January, when I do not doubt he will be found to have received the suffrages of the members. I have, therefore, to ask Mr. Bevan to take his place as one of our Hon. Secretaries.

Mr. Bevan said : I should like to thank you for the honour done me, and to assure you I shall do the utmost for the benefit of the Society at large and any of its members. I need hardly say how deeply I personally have felt the loss of our friend Mr. Davies. He was in every way a genuine friend, one whose opinion was always at the call of any member who chose to ask for it, and I can only feel it to be the more difficult to follow him ; but I trust, with your indulgence, to do my best.

THE ANALYST.

Mr. Cassal: I think, in view of the great loss which we deplore—for it is a very great loss to the Society, and one which will be felt for many years—that it would be a gracious and proper act if the Society itself were to pass a vote of sincere sympathy with Mrs. Davies and her family. This is a little more than a mere formal letter of sympathy, officially sent as from the Council. Having regard to the circumstances, I would suggest that a vote of sympathy be sent from this meeting of the Society.

The Chairman: I will take upon myself to propose that a letter of sympathy be sent by the Secretaries on behalf of the Society to Mrs. Davies on the occasion of the death of Mr. Davies. This, I am sure, will not require seconding.

On the motion of Mr. F. H. Perry Coste, seconded by Mr. Cassal, Mr. Chattaway and Mr. Kitto were re-elected auditors.

Dr. Dyer announced that the Council had nominated Dr. W. A. Tilden, the present President of the Institute of Chemistry, for election at the next meeting as an hon. member (applause).

Papers were then read on "An Improvement in Richmond's Milk Scale," by C. E. Cassal and B. H. Gerrans; and on the "Leffmann-Beam Method of Fat Estimation in Milk," Part iii., by H. D. Richmond and L. K. Boseley, and "On Vinegar," by E. Collens. The publication of these is unavoidably held over.

In the absence of the author, Dr. Dyer read the following paper:

ON THE ESTIMATION OF BEEF-FAT IN LARD.

By W. F. KEATING STOCK, F.I.C.

A QUALITATIVE process for the detection of beef-fat in lard is described in THE ANALYST for 1888 (p. 70). Its authorship is ascribed to Dr. Belfield, of Chicago. Probably every public analyst is acquainted with this test. Roughly, it consists in making a nearly saturated solution of a suspected sample in ether, and allowing this solution to stand in a test-tube loosely plugged with cotton-wool until separation of stearin occurs, after which the stearin crystals are examined microscopically, and the presence or otherwise of beef-fat is deduced from the crystalline forms observed. The whole procedure as set forth in THE ANALYST appears to be simplicity itself, and it is exceedingly simple when the operator has to do with a large proportion of beef-fat, but it becomes far otherwise when quantities of 5 per cent. to 7 per cent. are in question. Under certain circumstances it is possible to overlook the adulterant entirely.

The fact that there was no known method for the direct quantitative determination of beef-fat in lard was brought prominently before my notice in November, 1892, when a considerable number of samples of lard were submitted to me for analysis—all of which were normal as regards iodine-absorption, melting point of fatty acids, density at 99° C., and temperature-rise with strong sulphuric acid, but whose physical characters gave strong presumptive evidence of the presence of a solid fat which was not lard. Tested by Belfield's test for beef-fat as described above, good results were got in a few cases, but in others the presence of beef-fat was recorded as "doubtful," the crystallized ether-deposits failing to yield crystals having sufficiently well-defined

THE ANALYST.

beef-fat forms. This naturally led to experiment, and I sought first the temperatures at which beef-stearin and lard-stearin *began* respectively to separate from ethereal solutions of the same degree of concentration. I found that whilst beef-stearin began to deposit at 17° C., lard-stearin began to deposit at 14° C. I also found that the presence of lard and beef-fats in the same solution did not sensibly affect these temperatures. I satisfied myself further that, when Belfield's test failed with small proportions of beef-fat, it did so because the undesirable concentration of the ethereal solution resulted in a simultaneous deposition of lard and beef-stearin, and that interference, and not separation, was the consequence.

After making a large number of experiments, I selected 13° C. as the temperature at which I could best deal with commercial lards, and I adopted a *fixed* ether volume seven times greater than the volume of melted fat on which I operated. This, of course, involved the use of stoppered tubes instead of tubes plugged with cotton wool. I further recognised the necessity for bringing the solutions down to 13° C. as slowly as was compatible with convenience, and I fixed twenty-four hours as a time-limit. I may explain that the temperature of 13° C. was selected because it was obviously a guarantee of the deposition of any beef-stearin which might be present if lard-stearin was allowed to deposit, since, as shown above, the deposition temperature of beef-stearin is 3° C. higher than that of lard-stearin—as determined by myself. At this point I was struck by the idea of employing standard mixtures of lard and beef-fat, and, by means of comparison, obtaining quantitative results. The following process was the outcome of this idea :

PROCESS FOR THE ESTIMATION OF BEEF-FAT IN LARD.

This process depends upon the comparative insolubility of beef-stearin in ether at a temperature of 13° C. It is subject to modification according to the melting-point of the sample under examination. It must be employed with the discretion born of experience, and the procedure cannot be departed from in any degree if the author's results are to be criticised.

The requisites are six 25 c.c. graduated test mixers fitted with glass stoppers. A supply of ether, sp. gr. .720.

- "A." { Pure commercial lard melting at 34° to 35° C.
 { Beef-stearin melting at 56° C. (pressed beef-fat).
- "B." { Pure commercial lard melting at 39° to 40° C.
 { Beef-fat melting at 50° C.

A set of standard mixtures of "A" and a set of standard mixtures of "B" are prepared. They must contain 5, 10, 15 and 20 per cent. of beef-stearin and beef-fat respectively. The "A" set is used for samples melting at 33° to 39° C.; the set "B" is used for samples melting at 39° to 45° C.

The melting-point of all samples must be taken by the capillary tube method. The tubes must stand twenty-four hours after filling.

Suppose a sample which is to be tested for beef-fat melts at 34° C., 3 c.c. of the melted sample are run into one of the 25 c.c. test mixers, dissolved in 21 c.c. of the .720 ether, and placed in a vessel of water at 20° to 25° C. Three c.c. of each of the "A"

THE ANALYST.

series of standards are then dissolved in ether in exactly the same way. The five tubes are then cooled down to 13° C., and allowed to stand at that temperature or a lower for twenty-four hours. At the end of that time the apparent volume of deposit in each tube is noted, and this observation gives an immediate clue to the condition of the sample. The ether is poured off from the tubes as far as possible, and 10 c.c. of fresh ether at 13° C. added in each case. The stoppers are inserted and the tubes well shaken. When the deposit has settled out the operation is repeated. The whole contents of the tubes are now transferred to weighed shallow beakers. The ether is carefully run off (it may be used by repeated decantation to clear the tubes), but no fresh ether must be used. The deposits are dried at 40° C. for ten to fifteen minutes. The beakers are cooled and weighed. The standard weight nearest to that of the sample is used as the factor by which to calculate the beef-fat present.

The actual presence of beef-fat must be determined by the microscope. For this purpose a few particles of the dry residue are placed on a slide, moistened with alcohol and covered. *Very* moderate pressure must be applied to the cover, and the slide is viewed by a 1-inch objective and a C ocular. This power may, of course, be varied. With practice, however, there is no difficulty in recognising beef-stearin with the naked eye. The values of the standards being once determined, they need not be kept. The figures are constant if time, temperature and volume are constant.

Note.—The foregoing was written when it was perfectly easy to maintain a constant temperature of 13° C. for days. Since that time the author has had recourse to an ice chest, and he sees no objection to the tubes standing at a lower temperature than 13° C., provided that decantation and washing are carried out at 13° C. exactly.

Recent experiments have proved that by lowering the temperature of the filled tubes to about 16° C., and allowing them to stand two hours in water kept at that temperature by occasional additions of fragments of ice, and then standing them in water at about 10° C. over-night in a non-conducting apparatus, made of wooden boxes and dry sawdust, just as good results were got as when a constant temperature of 13° C. was available.

I may say here that I communicated the working details of this process to a number of my professional friends, and have received from them many assurances of approval.

I soon found that in my own practice the "B" set of standards met my wants, and I therefore give their values in the following table. But I would advise every operator to prepare the standards and determine their values for himself.

TABLE I.—COMPOSITION AND VALUES OF THE "B" SET OF STANDARDS.

Composition.		Melting-point Centigrade.	Ether-washed deposit in milligrammes.	Difference of ether-washed deposit.
<i>Beef fat.</i>	<i>Pure lard.</i>			
5	95	40.2	43	
10	90	41.0	81	38 milligrammes
15	85	41.6	122	41 "
20	80	42.4	181	59 "

The beef-fat melted at 50° C.

The lard melted at 39° C. and gave 8 to 11 milligrammes of ether-washed deposit.

The necessity for two sets of standard mixtures may not be obvious at first sight, but when it is seen how variable a substance commercial lard is, and when it appears that there are at least four kinds of commercial beef-fat, the necessity for *trying* to meet some of the problems likely to arise from these facts will likewise come in sight. The following table will perhaps best illustrate my meaning :

TABLE II.—TABLE OF DIFFERENCES FOUND IN PURE LARDS AND BEEF FATS.

Origin of sample.	Melting-point Centigrade.	Ether-washed deposit in milligrammes.
No. 1. Leaf lard (rendered in laboratory)	45·8°	146
No. 2. Belly lard " " "	39·0°	11
No. 3. Back lard " " "	34·5°	None in 24 hours
No. 4. Leaf lard (Liverpool merchant) ...	45·5°	114
No. 5. Soft lard " " "	34·0°	6
No. 6. Hard lard (Hull merchant) ...	42·5°	83
No. 7. " " (rendered by Mr. E. Bevan) ...	41·2°	90
No. 8. Pressed beef-fat (trade) ...	56·0°	Not determined.
No. 9. " " " "	53·0°	" "
No. 10. "Refined beef-dripping (trade) ...	43°	" "
No. 11. Hard beef-suet (rendered in laboratory) ...	50°	" "

The ether-washed deposit (E.W.D.) is, of course, the total lard-stearin deposited in each case, and it appears, as might have been expected, that the amount of E.W.D. is greatest where the melting-point is highest. But it further appears that when the stearin exceeds a certain proportion it is deposited in a rapidly-increasing ratio (compare Nos. 1 and 4). It will be seen also that although the melting-point of No. 2 is considerably higher than that of most samples of commercial lard, it gives only 11 milligrammes of E.W.D., whilst Nos. 3 and 5, with still lower melting-points, give none and 6 milligrammes of E.W.D. respectively. *These facts enable me to state the following proposition, That no normal sample of lard melting below 39° C. would give by my process an E.W.D. of more than 11 milligrammes.*

This, at any rate, has been my experience in applying my process to 80 samples of lard sent to me in my public capacity. Of these 80 samples, 32 have been found to contain beef-fat. In 27 of these cases prosecutions have been instituted, and in 25 convictions have followed. In two cases the summonses were withdrawn in consequence of conflicting certificates from the analysts at Somerset House. In both of the latter cases these gentlemen failed to find the adulterant. In one of them I sent the ether-washed deposit round to Mr. Alfred H. Allen, Mr. Otto Hehner, Dr. Alfred Hill, and Mr. E. W. T. Jones, all of whom certified to the presence of beef-fat stearin. Further comment is unnecessary.

By way of showing how the process has been applied by me, I append the results of the analysis of 21 of the adulterated samples :

TABLE III.

Origin of sample.		Melting-point.	E.W.D.	Result reported.
1.	Liverpool merchant ...	34.6° C.	50 milligrammes	7% beef-fat
2.	" " ...	34.6°	59 "	7% "
3.	" " ...	39.0°	52 "	6% "
4.	" " ...	45.5°	222 "	24% "
5.	Hull " ...	40.2°	73 "	9-10% "
6.	County sample, No. 68	34.6°	65 "	8% "
7.	" " No. 64	40.3°	102 "	12% "
8.	" " No. 394	37.6°	36 "	5% "
*9.	" " No. 455	38.8°	56 "	7% "
10.	" " No. 440	36.2°	38 "	5% "
11.	" " No. 260	38.6°	52 "	7% "
12.	" " No. 37	35.6°	34 "	5% "
13.	" " No. 7	36.7°	18 "	2.5% "
14.	" " No. 4	36.7°	39 "	5% "
15.	" " No. 18	41.8°	32 "	5% "
16.	" " No. 342	42.6°	178 "	20% "
17.	" " No. 340	41.8°	125 "	15% "
*18.	" " No. 8	37.6°	36 "	5% "
19.	" " No. 409	40.8°	54 "	7% "
20.	" " No. 265	41.5°	86 "	10% "
21.	" " No. 156	44.2°	151 "	18% "

The samples marked * are those in which the Somerset House analysts failed to find beef-fat.

Some of the figures I have given doubtless demand explanation. For example, it will be seen that in the table of pure lards and beef-fats (Table II.) I have shown that in two cases (Nos. 1 and 4) the E.W.D. rises to 146 milligrammes and 114 milligrammes respectively, and the question which is naturally prompted by intelligent criticism is: How would you proceed in a case where a sample, having the quality of Sample No. 4, had received the necessary addition of beef-fat to bring its melting-point and E.W.D. to about that of No. 1? Would the whole E.W.D. not be reckoned as due to beef-fat? Such a case demands careful consideration; but I have had the question to answer under very trying conditions when I was dealing with test samples, of the history of which I knew absolutely nothing. Due regard to the following points has enabled me to overcome any difficulty of this kind. 1st, The melting-point of the sample argues either a highly stearinized lard or a large proportion of beef-fat. 2nd, The *initial deposition temperature* serves as a most excellent guide to the proportion of beef-fat. 3rd, The bulk and character of the deposit, which forms before the temperature of 14° C. is reached, confirms the second observation. If the deposit at 14° C. is bulky and granular, it is due largely to beef-fat. 4th, The proportion of plumose crystals in the E.W.D. as seen under microscopic examination. If only 3 or 4 plumose crystals per field are visible, beef-fat cannot be present in large proportion. 5th, The E.W.D. is recrystallized from successive portions of 5 c.c. of ether, cooling down gradually to 14° C., until lard-stearin is no longer present in appreciable amount. The E.W.D. may now be looked upon as beef-stearin, and may be calculated into beef-fat of 56° C. melting-point. This was the course pursued

in Sample No. 4, Table III., where it will be seen that the melting-point is that of leaf lard nearly, whilst the E.W.D. is 222 milligrammes as against 146 milligrammes for leaf lard.

Direct experiment has shown me that neither cotton-oil, palm-nut-kernel-oil, nor cocoa-nut-oil interfere in any sensible degree with the deposition of the crystals of beef-stearin.

It would, I think, hardly be fair to those who may wish to employ the process I have described, if I left unnoticed some confirmatory tests which may be applied to the E.W.D. in cases of doubt, or if I failed to refer to some points of the microscopical manipulation.

It sometimes happens that the crystals of beef-stearin are thick and ill defined. In such a case recrystallization from 5 c.c. or so of ether is resorted to, and in my hands this has always effected the desired end. It is important that the solution should be slowly cooled as before. Petroleum spirit, having a boiling-point of 80° C., or under, may be employed for the same purpose, and gives good results.

The crystals should be mounted with care, and should not be subjected to any grinding motion in pressing down the cover-glass. It is, in my opinion, unwise to employ high powers for the examination of the stearin. The field is thereby restricted, and to my mind there is no substantial gain in definition after 100 diameters is reached. On the other hand, much may be learned by the use of the polarizer with selenites. Practice in these matters rapidly makes perfect. Some of the operators in my laboratory can speak to 5 per cent. or 7 per cent. of beef-fat in a lard by simply examining the E.W.D. with a pocket-lens.

DISCUSSION.

Dr. Dyer wished to know why beef-stearin, and not mutton-stearin, was always talked of. Was not the heavier portion of mutton fat used for the adulteration of lard, or was beef-fat only used for that purpose? He did not remember ever to have heard of a case where mutton-fat was alleged to have been added to lard, but he believed that both beef and mutton were used in the manufacture of margarine.

The Chairman observed that pig's fat was used in margarine only to a small extent, not mutton-fat. He had obtained a considerable number of deposits which were not beef-stearin and not pig-stearin. He (the Chairman) had had deposits which he was convinced were cotton-stearin. Mr. Stock had evidently gone much deeper into the question than any other public analyst in this country. The question was a very difficult one, so difficult that if any chemist had been asked, but a few years ago, what hope there was of distinguishing between pure pig-stearin or lard and beef-stearin, he would have answered that it was hopeless to expect to do so.

Mr. Allen said he had had the advantage of seeing the isolation of beef-stearin carried out in Mr. Stock's own laboratory, and he went through the experiments with him, Mr. Stock personally explaining every detail of the process. He spent much time over the microscope, pointing out the distinctions to be observed in various

mixtures of beef-fat with pig-fat or lard. He (Mr. Allen) was pleased with what he learned from Mr. Stock, and he was convinced he was very careful in coming to his conclusions, though he (Mr. Allen) confessed he had not succeeded in mastering all Mr. Stock's reasoning. The process was by no means a difficult one, and, as Mr. Stock had pointed out, fully justified his conclusion that beef-stearin was insoluble in ether at 13° C., and they would have next to no loss by crystallizing. He would recommend that the members of the Society interested in lard should give the process very careful attention. Mr. Stock he knew to be an admirable manipulator and of a very critical turn of mind, and he would not put before the Society these details without the strongest reason for his belief in them. He (Mr. Allen) had no doubt that when they had more experience they would be able to follow the manner in which Mr. Stock arrived at the figures, some of which at present appeared anomalous. It was a very gratifying fact that Mr. Stock's authorities had got convictions in most instances, and where they had not secured convictions, it was because Somerset House could not find beef-fat in samples which he (Mr. Allen) had personally examined and fully satisfied himself contained it.

Mr. Cassal had worked somewhat extensively at this subject, and could say that there was but little difficulty in detecting beef-fat so long as the work was done carefully and the procedure mentioned by Mr. Stock was followed. But he would be glad to have some further information from Mr. Stock as to his method of drawing quantitative conclusions. It was plain that Mr. Stock drew his conclusions from a consideration of all the data obtained, and that he especially relied upon the melting-points; but more definite instructions were requisite, and some further explanations were necessary in regard to some of the apparently abnormal figures for ether-washed deposit. Quantitative conclusions drawn from such figures as these would be, he presumed, settled mainly by the melting-points.

Mr. Allen said petroleum ether, as a crystallizing agent, gave a far better result than when ordinary ether was used.

(Conclusion of the Society's Proceedings.)

DISCUSSION ON THE VINEGAR QUESTION.

(Held at the Meeting, Nov. 1st, 1893.)

Mr. Alfred H. Allen, in opening the discussion, said: We are all very well aware of the interest and importance of the Vinegar Question at the present moment. I have already delivered myself of one or two opinions in print on this matter, added to which I have a great deal to say which is already stale to many of us who are reading various reports in the papers; but if I were not to mention, at any rate cursorily, the different points of interest in connection with the matter, I might deprive you of certain pegs on which to hang your arguments subsequently, and therefore I propose to go shortly over the history of the question from the commencement. In the first place, I may point out how many instances there are in the English language of words getting corrupted from their original meaning. Thus, geometry

does not now mean the measurement of the earth. We use the term trigonometry or mensuration to signify this. It is the same with vinegar, which we all know is derived from *vinaigre*, sour wine; but it has been diverted from its original application. No one would now contend that the term vinegar should be limited to wine which has undergone acetification. In certain parts of England the vinegar with which we are more familiar, made from malt or similar materials, is called "alegar." I contend that alegar is a perfectly parallel word to vinegar—that is, vinegar is sour wine and alegar is sour ale; and although ale does not make the best vinegar, alegar is a perfectly legitimate term, and it seems a pity it is not generally adhered to. By "vinegar" we do not now usually mean wine vinegar, though I have an analysis before me of a genuine wine vinegar. In many parts of the country by "white wine vinegar" is simply understood acetic acid. In that sense it has been perverted from its original meaning in a remarkable manner. A few months ago there was a considerable difference of opinion among analysts as to what should be included in the definition of vinegar. In the same manner we have no difficulty in saying that wine vinegar should be inside the definition, but there are certain articles which are very difficult to define. However, we have been relieved from that to a large extent by the decision at the Birmingham July Quarter Sessions, when the learned Recorder held distinctly that wood-acid was not vinegar. Therefore, we have now a knowledge of what is *not* vinegar, but the Court did not lay down exactly what vinegar was. The Recorder had before him a case of vinegar which was said by the public analyst to contain 70 or 80 per cent. of pyroligneous acid, and the defence admitted the accuracy of his certificate, while the actual analysis showed that this statement by no means over-estimated the proportion of pyroligneous acid present. The Recorder decided that wood-acid is not vinegar, although there were witnesses examined before him who contended that vinegar was anything which contained acetic acid of such purity and at such dilution as to be capable of being used for food. There is a great deal to be said for that view, but a great deal more against it. If we refer to our dictionaries, and works of special character bearing on the subject, we find almost invariably that "wood-vinegar" is distinguished from "vinegar" pure and simple, and hence it is not surprising to find that at Birmingham vinegar was defined in the following manner: "Vinegar is an acid liquid produced by the alcoholic and acetous fermentation of a vegetable juice or infusion." That is, perhaps, a little too stringent a definition, because there may be substances not coming within it which may nevertheless be held to be vinegar. When we once attempt to draw the line, the difficulty arises. Thus there is a German product made from distilled spirit, a product which varies to a certain extent in the details of its manufacture; this distilled spirit is taken and submitted to the acetic fermentation. By the fermentation acetic acid is produced, and acetic ether and other secondary products are also formed. That part of the process is such as is characteristic of the manufacture of a genuine vinegar. But distilled alcohol cannot be regarded as "a vegetable juice or infusion." A similar product is made by taking a malt wort or other saccharine liquid, and subjecting it to the acetous fermentation, and when that is complete, or nearly complete, a certain amount of alcohol is added to it, which in its turn is allowed to acidify, and gradually,

by repeating this treatment, a product is ultimately obtained containing 12 to 13 per cent. of acetic acid. Are we to regard this product as "vinegar," or should it not be sold always as "spirit-vinegar," or by some similar name, indicating its peculiar origin? In Scotland, distilled vinegar is preferred to undistilled, and is far more commonly met with. Are the people there not to have distilled vinegar, therefore, if they prefer it? or if they do, are they always to be obliged to ask for it as "distilled vinegar"? There are certain vinegar brewers who, recognising the importance of reducing the amount of albuminoids, and certain other constituents in the vinegar, distil a portion of their vinegar; then they add water to the undistilled portion, to obtain the weaker qualities, and add the distilled product. They claim that a vinegar so made will keep better, but at the same time they take away some important analytical distinctions between genuine vinegar and an article containing wood-acid. This practice has prevailed pretty extensively with certain vinegars, but it only applies to the very lowest grades of strength. That is a subject, therefore, which distinctly calls for our attention. It is very difficult, indeed, to draw the line as to what should be considered as genuine in such instances, and such mixtures are extremely liable to be mistaken for articles composed of fermentation vinegar mixed with wood-acid. In consequence of the acknowledged difficulty of distinguishing fermentation vinegar, where such additions had been made, from an article containing wood acetic acid, many of the inspectors who have submitted samples to analysts have been inclined to ask for samples of "malt vinegar." There are not a few manufacturers who advertise their products as malt vinegar, but "malt vinegar" is very rarely asked for by the public. They ask for Smith's vinegar or Brown's vinegar, giving the name of the manufacturer; but I believe it very rarely happens that people go and ask for "malt vinegar," and the result has been that a demand has been created for malt vinegar by the inspectors acting under the Sale of Food Act. There is no difficulty whatever where you have an adjectival prefix to the term vinegar. If a manufacturer sells his product as "pure wood vinegar," or "pure unfermented vinegar," which is the description adopted by a certain vinegar "faker," I do not know that we can complain of it. But I think that it is a dangerous practice to say that a vinegar contains a certain amount of "pyroligneous acid," unless the presence of tarry products makes its origin absolutely certain. If my information is correct, the term pyroligneous acid is applied in commerce to a particular article which has not had the advantage of being purified from the tarry products which are usually associated with crude pyroligneous acid. When purified by conversion into a lime or soda salt I believe it ceases technically to be called "pyroligneous acid," and is denominated "acetic acid." I have in my mind a case where an analyst, called for the defence, said that a vinegar did not contain pyroligneous acid because there were no tarry products present. That was probably perfectly correct. The term pyroligneous acid may indicate the origin of an acetic acid, but unless tarry products are distinctly recognisable it is in my opinion a mistake to use the term. I believe it is the fact that vinegar is now never, or hardly ever, made from malted grain alone. I believe there is practically no vinegar now made solely from malted barley; there is always a mixture of unmalted barley, or other unmalted grain with it. Public analysts have been inclined to say, "Yes,

that is all right; we understand by malt vinegar, a vinegar made from grain where the starch has been converted into saccharine matter through the action of diastase, and, seeing that the diastase in malt is greater in quantity than is necessary to convert the starch of that malt, by converting the starch of unmalted barley into sugar you are merely making the diastase do its full duty." But if, accepting the above reasoning as correct, we give as a definition of "malt vinegar," "vinegar made from saccharine matter which has been converted from starch purely through the action of diastase," we are immediately met with the question, "Why limit the converting agent to diastase?" The diastase converts the starch into a mixture of maltose and dextrin. Why should we be limited to diastase? Why may not we use saliva? Why should a manufacturer be limited to diastase if he can accomplish the same result with another ferment or by another substance capable of producing hydrolysis? Why should not he convert the starch into malt sugar and dextrin by means of acid? I have here cut, from the *Brewers' Journal*, a whole series of advertisements of various preparations which the vendors call "malt." The general practice in the brewing trade appears to be to regard as "malt" any saccharine material produced from grain, either by a process of cooking, by the action of diastase, or by conversion with acid, the result being the same, though the tools employed are different.

It is interesting to consider the definitions of malt given in our standard dictionaries. Thus Johnson describes malt as "Grain steeped in water, and fermented; then dried on a kiln."

Ogilvie's Imperial Dictionary, which is a modern one, says that the word malt is derived from the Anglo-Saxon *meltan*, to melt, to dissolve, to digest, to cook; and defines malt as "grain, usually barley, steeped in water and made to germinate, the starch of the grain being thus converted into saccharine matter; after which it is dried in a kiln, and then used in the brewing of porter, ale, or beer, and in whisky distilling."

The new edition of Webster's Dictionary defines malt as "barley or other grain, steeped in water and dried in a kiln, thus forcing germination until the saccharine principle has been evolved. It is used in brewing and in the distillation of whisky."

In other words, two of our modern standard dictionaries stick to the definition of malt as meaning sprouted grain, and do not give the term the extended meaning which beer-brewers do, and vinegar-brewers would like to, put upon it. My own feeling is that the public analyst stands between the public and the vendors of these articles. What does the public understand by malt? An intelligent man desiring information would turn to a dictionary and would say: "I see, it is made from grain which has sprouted." Therefore, the public would expect, if they asked for malt vinegar, that it would be made from such sprouted grain. On the other hand, we cannot at present recognise any chemical distinction between acid converted and diastase converted starch. But it does not follow that there no difference exists. It is quite possible that there are distinctions which we have hitherto failed to discover, just in the same way as there are distinctions between the different qualities of whisky and other fermentation products which are at present inap-

preciable to the most careful analyst. As chemistry advances we may be able to ascertain in what these distinctions consist. The manufacturer says that the term malt means to melt, and that whether they melt or dissolve it by means of acid, or diastase, or saliva, it makes no difference. Besides, it is argued that this diastase is, after all, only a solvent, and when it is done with you want to get rid of it, as the albuminoids are very objectionable. I believe vinegar manufacturers do attempt to keep down the albuminoids and use processes more or less suited for eliminating a portion of the phosphates and other substances which are apt to facilitate secondary changes. It is quite possible that vinegar keeps better and is otherwise materially improved if the conversion of the starch is effected by the agency of acids instead of by diastase. I am stating here the arguments of intelligent technical brewing men, because they are arguments which will most probably be used in important cases in which public analysts will be concerned in the future. I have already stated that, to my mind, whether we go to the dictionary definition or not, we should protect the public by requiring that the conversion should be by diastase; but, on the other hand, if it be held that we are drawing the rein too tight by requiring that malt vinegar should be made through the action of diastase, then it becomes a question of what is desirable. We have in vinegar brewing a legitimate industry, carried on with a certain amount of scientific knowledge which may be very greatly improved on, but in which there is a certain amount of skill and ability, and the vinegar brewers are producing a good merchantable article, which, although it may not satisfy the requirements of public analysts, is in a very different position to the product of the vinegar "fakers," who, by the aid of a second-hand tub, and a few barrels of acetic acid, set up as vinegar "manufacturers," and produce an article which is very different in character from a genuine brewed vinegar. Vinegar varies as much in quality as does wine, and there is a taste to be cultivated in vinegar just as there is in wine. I should like as far as possible, legitimately with our duties as public analysts, to see the trade in vinegar encouraged, instead of our giving countenance to German manufacturers who import their product as "acetic acid"; and then, having coloured it to represent vinegar, sell it in opposition to those who make vinegar by true fermentation processes. I am sorry to think that sometimes the manufacturers, for whom I have individually a great respect, have been rather hardly dealt with by public analysts who have not always taken into consideration the whole of the circumstances of the case. It has become necessary for us to discriminate between the different kinds of vinegar, and the question arises, How are we to do it? Unfortunately, we are at sea for want of definite information as to the composition of vinegars of various characters; and, seeing that these different kinds of vinegar overlap at each stage, and manufacturers are perfectly justified in improving their vinegar by taking out the constituents which are liable to cause an objectionable change in their product, however inconvenient such practices may be to public analysts, it follows that we are necessarily in a very great difficulty.

The limited quantity of vinegar ordinarily brought to a public analyst is another practical difficulty with which we have to contend. I am sorry to say that some of my inspectors have, on occasions, brought me one-third of a pennyworth. There is no object in purchasing so small a quantity as half a pint, or even one pint. It

would be much more sensible to buy a barrel, as was actually done in one instance at the suggestion of the analyst. Really the question of using 100 c.c., or 1,000 c.c., is not to the purpose if we can ascertain something which we could not ascertain with smaller quantities. The succinic acid of vinegar I hope to be able to find useful to us in the future, although I have nothing very favourable to report at present, and am now trying if the glycerin is of any value as a proof of origin. Alcohol always exists in a well-made fermentation vinegar, for manufacturers stop the process before the acetification is complete. I have some instances of vinegar which has diminished in strength to the extent of fully 1 per cent. of acid in six months. If the alcohol is all destroyed the change is likely to be much more rapid afterwards, since, in the absence of other food, the vinegar-fungus feeds on the acetic acid previously formed. A well-made vinegar should contain alcohol, not only for its keeping purposes, but also because you have a gradual formation of acetic ether, just the same as in wine after keeping. We might distinguish the fermentation vinegar in that way. At the same time we must remember that it is very easy to add alcohol in imitation of a fermentation vinegar. The German manufacturers put acetic ether into their acetic acid with a view of making it as like vinegar as possible. There is a considerable amount of solid extract in fermentation vinegar, but when you are dealing with a mixture containing pyroligneous acid the quantity is very much less. The solid matter varies very much according to the perfection of the fermentation, and affords an indication of some value, though not so great as the amount of ash, which does not vary to a great extent through the fermentation. The proportion of sulphuric acid will afford some information as to the probable use of glucose, and so on. We have carried out the estimation of nitrogen on a large number of samples, and find it to give us a very valuable criterion. First of all, you cannot get grain vinegars without a large amount of nitrogen; for although the manufacturers do attempt to remove a certain amount of the nitrogenous matters, there is always a large amount left. In estimating the total nitrogen by the Kjeldahl method, we did not add sulphuric acid direct to the vinegar, as is the custom of some analysts, but have evaporated the vinegar to dryness, or at any rate to a syrup, before adding the acid. 25 c.c. of vinegar is a convenient quantity to employ. The nitrogen found can then be calculated to its equivalent of albuminoids by the usual factor; but I am much inclined to think that much of the organic nitrogen of vinegar exists as peptones or similar soluble forms. In one case where we made the experiment one-tenth of the whole content of nitrogen was found to consist of ammoniacal salts. The proportions of all these constituents must necessarily vary with the strength of the vinegar. A wort which originally contained 12 per cent. of sugar and other solids will necessarily contain more nitrogen, ash, phosphates, etc., than a vinegar which originally contained only 7 per cent. of sugar. Therefore, it is extremely desirable to adopt Mr. Hehner's plan of calculating the various constituents upon the original solids of the vinegar; 60 parts acetic acid are theoretically produced from 90 of glucose, and hence, if we multiply the acetic acid found by 1.5, we obtain the amount of sugar from which that acetic acid was derived.

Adding to the figure thus obtained the total extractive matters still contained in the vinegar, we obtain a number representing the "original solids" of the wort.

Thus, if a vinegar contain 5.2 per cent. of acetic acid and 2.8 of extract, the original solids will be $5.2 + 2.6 + 2.8 = 10.6$. If the vinegar itself contained 0.08 of nitrogen, the original solids will contain—

$$\frac{0.08 \times 100}{10.6} = 0.75 \text{ per cent.}$$

In this manner one can eliminate the differences caused by variations in the strength of various samples of vinegar, and reduce the results to a kind of common denominator. As a matter of fact, the loss of acetic acid in the process of manufacture averages some 30 per cent., so that the proportion of original solids calculated in the above manner is always below the truth. Hence a nearer approximation to accuracy would be obtained by multiplying the acetic acid by 2.25, instead of 1.5, before adding the extract. But the change would involve confusion, and hence I have adhered to the mode of calculation originally suggested by Mr. Hehner.

During the last few months upwards of 200 samples of vinegar have been analysed in my laboratory. Many of these were samples submitted under the Sale of Food Act, but a considerable number were typical samples of vinegar submitted by leading manufacturers. It is useless to distinguish the samples of "vinegar" from those of so-called "malt-vinegar," as many of the latter were found on analysis to be very far from justifying such a description.

I must leave for some future occasion the question of the range of variation in composition possible in pure vinegars of various origins, but give on the opposite page the figures representing, according to my experience, the composition of certain typical vinegars of definite origins.

Of the samples of which the quantitative analytical results are given above, the first four, with the possible exception of A, I believe to be genuine grain vinegar, brewed from a mixture of malted and unmalted barley or similar grain, and the starch converted entirely by the action of diastase. E is the average analysis of the first seven vinegars of Hehner's table (*THE ANALYST*, xvi. 82), described by him as "partly to my knowledge undoubtedly made from malt only, partly made by the best London manufacturers."

F and G are genuine vinegars brewed from a mixture of malted and unmalted barley, with the addition of sugar.

H and I are vinegars manufactured chiefly from rice, the conversion being effected by the aid of sulphuric acid. They contained respectively 0.138 and 0.143 grammes of combined SO_3 per 100 c.c.

J. and K were manufactured from sugar, with perhaps a little malt in the former case.

L is the vinegar condemned by Dr. Hill, which formed the subject of the appeal before the Birmingham Recorder. The figures have been communicated by Dr. Hill.

M is a sample of very pale vinegar, made by mixing distilled vinegar with a little of the same vinegar undistilled. It possessed a very appetising smell and taste.

(To be continued.)

Sample Mark.	A	B	C	D	E	F	G	H	I	J	K	L	M
Specific gravity ...	1.0205	1.0170	1.0228	1.0160	—	1.0130	1.0185	1.0190	1.0160	1.0104	—	1.0070	1.0104
Per 100 parts of vinegar													
Acetic acid ...	6.61	6.39	5.26	4.86	4.23	5.22	5.82	5.58	5.70	3.51	4.92	4.70	7.00
Total solids ...	2.81	2.67	3.96	2.31	2.70	1.56	2.45	2.98	2.09	1.52	1.76	0.21	0.10
Ash ...	0.55	0.34	0.40	0.47	0.34	0.30	0.39	0.30	0.43	0.27	0.278	0.04	0.015
containing:													
Alkalinity as K_2O	0.102	0.091	0.118	—	0.024	0.03	—	0.013	—	0.080	—	Trace.	Trace.
Phosphoric acid ...	0.066	0.077	0.093	0.057	0.105	0.064	0.041	0.017	0.024	0.010	0.016	0.009	None.
Nitrogen ...	0.120	0.099	0.095	0.099	—	0.052	0.037	0.104	0.052	0.014	0.016	—	0.002
Albuminoids ...	0.756	0.624	0.598	0.624	—	0.328	0.611	0.655	0.390	0.088	0.103	—	0.013
"Original solids"	12.73	12.26	11.85	9.60	9.35	9.39	11.18	11.35	10.64	6.81	10.02	7.26	10.60
Per 100 parts of original solids:													
Ash ...	4.32	2.78	3.37	4.92	3.64	3.20	3.49	2.64	4.04	3.94	2.77	0.55	0.14
Phosphoric acid	0.52	0.63	0.79	0.60	1.16	0.68	0.37	0.15	0.235	0.14	0.16	0.120	None.
Nitrogen	0.95	0.816	0.80	1.03	—	0.56	0.87	0.93	0.553	0.206	0.16	—	0.019
= Albuminoids	5.98	5.14	5.04	6.49	—	3.53	5.48	5.86	3.670	1.30	1.03	—	0.120

* The figures are grammes per 100 c.c. of the vinegar.

THE PROXIMATE COMPOSITION OF BUTTER.

BY H. DROOP RICHMOND.

At the end of 1890 Dr. Vieth read a paper before the Society, in which he gave in abstract the results of 267 analyses of butter, and described the method used for these (ANALYST, xvi. 1). Since that time samples have been regularly examined by Dr. Vieth up to March, 1892, and since then by myself by the same method.

In view of the interest now attaching to the question of the amount of water in butter, I have thought it desirable to supplement Dr. Vieth's paper by giving the figures obtained up to the present date. I have nothing to add to his remarks on the methods of churning and preparing butter for the market, nor to the details of the method of analysis given in his paper.

In Table I. are given the results of the whole of the water determinations made by Dr. Vieth and myself; this is a simple extension of that given by Dr. Vieth (*loc. cit.*), and is in the form adopted by him.

TABLE I.

Percentage of Water.	Number of Samples.	Per cent.
7-8	2	.4
8-9	8	1.4
9-10	18	3.2
10-11	37	6.6
11-12	76	13.6
12-13	95	17.0
13-14	210	37.5
14-15	88	15.7
15-16	21	3.8
16-17	3	.5
17-18	2	.4
	560	100.1

A reference to the table given in Dr. Vieth's paper shows that, although the number of samples is more than doubled, the percentages of water found in butter is practically unchanged, the third columns (percentages of total samples) differing by only 1.5 per cent. as an extreme.

These 560 samples include various kinds of butter, among them being English, French, Danish, Swedish, German (Kiel), and Australian butters; many of them were churned at Bayswater, practically speaking, under the eyes of Dr. Vieth and myself, while the others were samples of butter on the market. As it is a well-known fact that butter loses water during the handling it receives in commerce, I have thought it desirable to divide these samples into two classes—English butters, which were analyzed soon after churning; and foreign butters, which had stood a sea-voyage and been otherwise handled.

TABLE II.—ENGLISH BUTTERS.

Percentage of Water.	Number of Samples.	Per cent.
7-8	2	1.4
8-9	5	3.5
9-10	13	9.1
10-11	17	11.9
11-12	36	25.2
12-13	33	23.1
13-14	23	16.1
14-15	9	6.3
15-16	4	2.8
16-17	1	.7
	143	100.1

TABLE III.—FOREIGN BUTTERS.

Percentage of Water.	Number of Samples.	Per cent.
7-8	—	0
8-9	3	.7
9-10	5	1.2
10-11	20	4.8
11-12	40	9.6
12-13	62	14.9
13-14	187	44.9
14-15	79	18.9
15-16	17	4.1
16-17	2	.5
17-18	2	.5
	417	100.1

The history of the English butters is known, and they were made with a reasonable amount of care, as commercial butter; nothing is known of the origin of the foreign butters, and these may have included samples purposely mixed with water or which, by carelessness in manufacture, contained an excess of buttermilk. Dr Vieth has expressed the opinion that 16 per cent. of water should be considered as the maximum allowable in butter, and considering the above tables, I can only give my heartiest approval of this view.

Out of the whole 560 samples, but five of them contained more than 16 per cent of water. One of these, a Swedish butter, contained a great excess of curd, and had a cheesy taste, and was evidently, from this fact alone, a very carelessly-made butter; another sample, the only English one above 16 per cent. (contained 16.4 per cent.), was churned at a very high temperature, owing to the weather being very hot, and a supply of ice temporarily not obtainable. Of the other three samples, one was a Danish butter, and the other two Kiel butters.

In my opinion, the adoption of 16 per cent. as the highest permissible limit will inflict no hardship on honest traders, as it is quite high enough to include butters in

the churning of which slight errors of judgment or mistakes—to which even the best dairyman is liable—have been made, while the adoption of higher limits will but open the way to fraudulent addition of water.

WATER ANALYSIS—THE INTERPRETATION OF RESULTS—*Continued.*

BY F. WALLIS STODDART.

(Reprinted from "*The Practitioner.*")

THE one flaw in both limits is that neither has any better foundation than general experience, which has not invariably supported them. There is a wide difference, however, between these definitions of purification, and that based simply upon the completion of nitrification. Is either limit to be accepted as satisfactory? And if so, which? Convincing answers to these very important queries are to be obtained only by direct experiment as to the conditions of nitrification, and a consideration of recorded cases of the spread of disease by polluted water. What is required is fairly exact information as to the extent both of space and time within which nitrification can be completed, and the chances of survival of pathogenic organisms under the same condition.

(a) With regard to the depth of soil, and the length of time within which nitrification can be completed, two methods of investigation are possible: the artificial production of nitrification, and the examination of natural waters under known conditions.

Now, in connection with the first method some very remarkable experiments were recorded by the Rivers Pollution Commission in their First Report. In these experiments sewage was filtered through soils of various kinds contained in glass cylinders, and the very noteworthy result was obtained that, using a soil in which the process of nitrification was thoroughly established, no less than 97 per cent. of the nitrogen of London sewage could be converted into nitrates by percolation through only five feet of soil.

There are, however, some points in which these experiments might be improved, in the light of more modern knowledge as to the character of the nitrification process. For instance, the sewage was poured on the soil twice every twenty-four hours, thus alternately waterlogging the soil and leaving it comparatively idle. On the other hand it might be objected that the filtrate was collected only once a week, and that the process of nitrification might be, and probably was, proceeding all the time in the water. Still it is sufficiently remarkable that in these experiments raw sewage was converted into a water which compared favourably as regards organic matter with that supplied to the Metropolis at that date (1869) for domestic purposes.

I have for some time adopted a similar arrangement for class demonstration, using columns of coarsely-powdered chalk about two inches in height contained in glass tubes. By carefully seeding these tubes with the appropriate organisms, it is easy to produce quite a copious formation of nitrous acid from ammonia, or nitric from nitrous acid, by allowing the proper solutions to drop slowly upon the upper surface of the chalk.

I have more recently extended these experiments in order to get quantitative results. My filter consists of a column of coarsely-powdered chalk twenty-four inches in height contained in a one-inch condenser tube. A drop arrangement is made so that the water trickles slowly but continuously over the particles of chalk, thus representing as nearly as may be the conditions existing in the superficial air-charged layers of soil. Nitrification is established by first dropping a little putrid urine on the chalk and then passing a solution of urea (0·01 per cent.) until the effluent is strongly ammoniacal. A polluted well-water in which the nitrous fermentation predominates is then substituted for the urea, and when nitrites appear freely, a strongly nitrating well-water is used in the same way. For some reason I have succeeded better with well-water than with extracts of soil. If now a solution of ammonium carbonate, containing 1 part nitrogen in 10,000 and representing sewage, is allowed to flow through the tube, a very constant formation of calcium nitrate is observed. By suitably forcing the rate of flow, nitrite and ammonia may readily be made to appear in the filtrate; but a very considerable quantity of solution can be completely nitrated daily.

My first experiment with this filter was made with a very badly polluted well water, which, however, was perfectly bright and contained no suspended matter; it was passed through at the rate of about one pint in twenty-four hours, and afterwards at double this rate. The results given in the following table expressed in grains per gallon were obtained.

Here we have a water which would be condemned unhesitatingly as rankly polluted, and beyond question dangerous to health, so altered in composition as exactly to resemble what I have frequently seen described by analysts who confine themselves to a determination of the "ammonias" as "water of remarkable organic

	Original water.	Filtered water.	
		1 pint per diem.	2 pints per diem.
Saline ammonia	·252	·0063	None
Albuminoid ammonia	·018	·0023	·0056
Nitrogen as nitrates and nitrites ...	4·61	5·77	5·48
	(Nitrites abundant)	(No nitrites)	(No nitrites)
Chlorine... ..	8·8	8·8	8·9
Total solids	112·0	106·0	107·0
Phosphoric acid	Very heavy trace	...	Very heavy trace
Total hardness	57·0	...	56·5
Permanent hardness	33·0	...	39·0
Oxygen absorbed in fifteen minutes ...	·084	...	·034
Oxygen absorbed in four hours ...	·112	...	·059
Colour	Deep yellow-brown	...	Yellow-green

purity," and this by a natural process which could not possibly be imagined to ensure the removal of infective matter. The second sample yielding a quart per diem represents about the maximum amount of impurity which could pass muster without

remark, such water being frequently described within my experience as "slightly contaminated with vegetable matter (absence of saline ammonia), and somewhat highly charged with mineral salts of an innocent character." Now, this interpretation is plainly entirely beside the truth, and a proper appreciation of the results other than the "ammonias," chlorides, and total solids, even if the effluent were largely diluted with pure water, would at once lead to a recognition of the dangerous character of the supply.

Two interesting points are incidentally brought out in this experiment—the increase of permanent at the expense of temporary hardness, and the abundance of phosphates in the filtered water, of which 70 ccm. under suitable treatment gave a decided precipitate with molybdate, thus showing that there is no foundation for the assertion that carbonate of calcium and phosphates are incompatible in drinking water.

The water was travelling through the filter at the rate of two feet in about forty minutes, a rate probably considerably in excess of that at which subsoil water generally moves.

It may be objected, however, that nitrification was already far advanced in this water when treated, implying a certain unknown amount of previous natural filtration; besides, it is desirable to know how rapidly raw sewage is affected. I therefore procured crude sewage, and finding one tube did not carry nitrification to sufficient completion when keeping up the same rate of flow, I added another, bringing up the total height of the column to five feet. I may point out that my object in making this modification, instead of diminishing the rate of flow, was to procure sufficient water for analysis without leaving it to accumulate more than twenty-four hours; the same end might be arrived at by duplicating the apparatus, but five feet of soil did not seem an unreasonable distance to assume as generally intervening between well and sewer. The nitrification with this filter improved constantly for weeks, and when the process was stopped the filter was yielding water with the following composition at the rate of one pint per diem:

	Sewage.	Effluent water.
Saline ammonia	3.85	.0013
Albuminoid ammonia175	.0158
Nitrogen as nitrates	None	5.99
Nitrites	None	None
Chlorine as chlorides	7.9	7.8
Oxygen absorbed in fifteen minutes566	.066
Oxygen absorbed in four hours	1.22	.131
Total dissolved solids	51.0	79.0

The sewage then was converted, by percolation through five feet of coarse soil, into water which if not of completely satisfactory organic purity as judged by the "ammonias" only, is certainly as pure as the well-water which in a previous experiment was improved into a first-class water by further percolation through two feet of similar soil: and if the sewage effluent be supposed to leak into a well of unpolluted water, it may form at least one-third part of the whole supply without

being detected by this test. This experiment then more than confirms the experience of the River Pollution Commissioners—I say more than confirms, because the rate of flow is enormously greater in my form of experiment, one cubic yard of soil dealing more completely with 155 gallons of sewage per diem, than the same quantity does with ten gallons in that of the Commissioners. This is obviously due to the more regular and continuous flow combined with perfect aëration: and it is fair to assume that the process will be completed even more rapidly in the natural soil, especially in that of large towns, where it has been in constant operation for generations.

A simple calculation shows that the flow of one pint of water per diem through a filter one inch in diameter corresponds to a yield of twenty-three gallons per square foot; or in a well twenty feet deep and three feet in diameter, a yield of over 4,000 gallons a day. This is sufficient to account for the fact that the prevailing type of shallow-well-water in large towns is that showing oxidized pollution, and it is only where the source of pollution is in very immediate contiguity to the well that much unaltered sewage is found in the water. In such cases it is commonly the case that other wells in close proximity show no signs of such unoxidized sewage. I give here an instance of two such wells belonging to adjoining houses, which illustrates this point sufficiently.

More direct evidence, however, is afforded by waters contaminated from a source of pollution the position of which is known. The third example in the table is a case in point, where a difference of opinion as to the purity of the water was cleared up

EXAMPLES OF RAPID NITRIFICATION.

	Well to well, 10 yards.		Drain to well, 8 yards.
	A.	B.	
Saline ammonia	·018	None	·0004
Albuminoid ammonia	·020	·005	·0035
Nitrogen as nitrates and nitrites ...	6·92	4·61	4·04
Nitrites	Abundant	None	None
Chlorine	10·8	7·8	15·05
Oxygen absorbed in fifteen minutes ...	·081	·028	—
Oxygen absorbed in four hours ...	·126	·070	0·45
Total dissolved solids	142·0	116·0	103·2
Total hardness	75·0	62·0	40·9
Permanent hardness	51	38·0	—
Appearance, etc.	Good	Good	Good

by the discovery of a choked drain at a distance of about three yards, with evident signs of percolation into the well.

A number of similar examples will be found in THE ANALYST, viii., p. 59.

The necessary extent of percolation for the practical completion of nitrification is then limited to at most a few yards, a distance quite insignificant when compared with that which has been shown in many epidemics to fail entirely as a protection against the conveyance of disease.

As to the duration of this process, a very false impression has, I think, arisen

owing to the majority of investigators having carried out their experiments in flasks, whereby the operation is much retarded in consequence of insufficient aëration. Under suitable conditions, as already shown, it occupies only an hour or two with raw sewage.

(b) Few persons, I take it, would rely on filtration under the above circumstances to remove specific organisms by mechanical retention. The experiments of Plagge, Piefke, Koch, and others, have shown that carefully prepared artificial filters can only effect a partial purification when the whole of the conditions are under control, and that no filter can be relied upon to effect even this for an unlimited time. I find that the filter tube above described does produce certain interesting effects upon the various forms of micro-organisms abounding in sewage, but is totally inefficient in preventing the passage of the cholera spirillum. A few drops of a culture introduced into the sewage passing into the filter yielded abundant evidence of the presence of the spirillum in the effluent after an interval of about two hours, and continued to do so for some hours—until, in fact, the filter was dismantled for sterilization.

There are, however, one or two other points worth consideration, in particular the length of time pathogenic organisms can persist in natural waters. A great number of investigators have worked at this question, and their results are conveniently summarized by Professors Frankland and Marshall Ward (*Proc. Roy. Soc.*, li. 183). These results are not very concordant, obviously in part because of varying conditions of experiment. All, however, are agreed that the cholera spirillum will persist in fairly pure water for several days, especially if in vigorous condition, and that probably the typhoid bacillus is even more resistant. I am convinced that this estimate, though abundant for our present purpose, is much understated.

I have been experimenting with the cholera spirillum (as being very sensitive and easily recognised) for some time, and may briefly state here that there is no difficulty in keeping it alive at the ordinary temperature for some weeks in pure and in polluted well-water, and for a still longer time in distilled water and various sterilized saline solutions. I attribute the low estimate of some observers to a want of appreciation of the fact that no culture process probably represents adequately the conditions of the animal body, and that certainly the gelatine plate process is very far from doing so. By cultivating at the body temperature in broth or peptone-salt solution, typical growths may be obtained not only from material apparently exhausted, but also from polluted waters which give hopelessly complicated results with the gelatine plate. However, as nitrification occupies a few hours only, there is no reason to doubt the persistence of pathogenic forms on the score of time.

V. Nor is there satisfactory reason to suspect such antagonism between water organisms and pathogenic forms as to lead to rapid destruction of the latter.

Very little appears to have been done in the direction of allowing pure cultures to grow in the same solution. Professor Marshall Ward, however, shows in the second Report to the Water Research Committee of the Royal Society (*Proc. Roy. Soc.*, liii. 164) that anthrax gets the better of the bacillus fluorescens under these conditions; but as mentioned above, I have found the cholera spirillum persist for many days in both classes of polluted water, *i.e.* waters containing much fresh sewage and those in which nitrification is nearly completed, and in the presence, therefore, of

innumerable organisms of the various kinds naturally present in such water; and further that it will pass through soil in which sewage is nitrifying rapidly without apparent hurt. The only ground, therefore, for suspecting a destructive influence on the pathogenic forms on the part of water organisms being the apparently greater persistence of the former in sterilized than in non-sterile waters, a result which may not unlikely be due in part to defective methods of examination, I conclude that such influence is not a very powerful one, if it exist at all. A great point is made of this antagonism in the recent Report of the Royal Commission on the Water Supply of the Metropolis, but no new positive evidence is adduced in support of the theory.

(To be continued.)

The Determination of Bromine in Urine. A. Nicolle. (*J. Pharm. Chim.*, 1893, xxviii. 298; through *Chem. Zeit.*)—Most processes in vogue for the determination of bromine in urine, in presence of chlorine, are tedious and operose, as, for example, the conversion of the mixed precipitate of silver chloride and bromide into chloride by ignition in a stream of chlorine. The volumetric estimation by titration with chlorine water, and the absorption of the liberated bromine in carbon disulphide, has two disadvantages, viz., the necessity of daily standardization of the solution of chlorine, and the difficulty of discerning the end point. The author therefore adopts Dechau's process, depending on the use of potassium bichromate.

50 c.c. of the urine are evaporated to dryness, with the addition of 2 grammes of caustic potash, and incinerated at a low red heat. The ash is extracted with boiling water, the solution filtered, and the filtrate—which should be colourless, and measure with the washings about 40 c.c.—treated with 10 c.c. of sulphuric acid and poured into a flask containing 20 grammes of potassium bichromate. The flask is connected with a bulb tube containing 20–25 c.c. of a 4% solution of potassium iodide, the apparatus being preferably of glass throughout, or (if rubber must be used) with the rubber connections freed from excess of sulphur by previous extraction with caustic potash. The contents of the flask is heated to boiling for a quarter of an hour, the contents of the bulb tube diluted to 50 c.c. and titrated with sodium hyposulphite. In the presence of iodides in the urine, the aqueous extract of the ash is only neutralized with sulphuric acid and distilled with potassium bichromate, under which conditions the iodine alone is liberated, the bromine being afterwards obtained on adding a further quantity of sulphuric acid and continuing the distillation in the manner already described.

The presence of sulphur in the urine, notably when it contains albumin, may cause trouble by the formation of sulphides, which are dissolved by the extraction of the ash, and interfere with the estimation of the bromine by the action of the sulphuretted hydrogen to which they give rise. This can be met by adding oxalic acid to the aqueous extract of the ash, and boiling until no more sulphuretted hydrogen is given off. When only sulphates in the urine have to be provided for, precipitation of the original liquid with barium chloride suffices. B. B.

NOTE BY ABTRACTOR.—To avoid possible liberation of halogen acids by oxalic acid, the use of succinic acid (*THE ANALYST*, 1893, 255) may be applicable here.

The Determination of the Specific Gravity of Curdled Milk. **M. Weibull.** (*Chem. Zeit.*, 1893, xvii. 1679.)—The author adds a known volume of ammonia to the curdled milk, mixes well, and determines the specific gravity of the mixture. As the specific gravity of the ammonia is known, and no sensible alteration of volume occurs on mixing, the specific gravity of the milk is easily calculated. The following table shows the accuracy of the method when tried on samples the specific gravity of which was taken while they were fresh, and then again after the lapse of a few days.

Sp. Gr. Fresh Milk.			Days kept.			Sp. Gr. calculated from that of Milk and Ammonia.
1.0243	10	1.0243
1.0243	10	1.0243
1.0154	12	1.0151
1.0317	4	1.0320
1.0283	4	1.0280
1.0217	1	1.0217

B. B.

The Volumetric Determination of Alkaloids in Drugs. **C. Caspari and A. Dohme.** (*Pharm. Rundsch.*, 1893, xi. 229; through *Chem. Zeit.*)—The conventional method for the determination of alkaloids in drugs consists in extracting them in a state of approximate purity, and weighing the substance thus obtained. The author, in common with other chemists, considers that the approximation to purity usually attained leaves much to be desired, and prefers the volumetric method. His strength of conviction may be gathered from the following dicta:

(1) The volumetric method for determining alkaloids in drugs by means of standard acid is the most reliable and exact method with which we are at present acquainted.

(2) Gravimetric results, which are stated as representing the contents of pure alkaloid, are in most cases erroneous.

B. B.

The Chemical Recognition of Horse-flesh. **W. Brautigam and Edelmann.** (*Pharm. C. H.*, 1893, xiv. 557; through *Chem. Zeit.*)—The method is based on the use of the well-known iodine reaction of glycogen, a body which is a constant constituent of horse-flesh. The finely-divided flesh is boiled with four times its weight of water, and the resulting broth treated with dilute nitric acid, to precipitate albuminoids, and filtered. Saturated hydriodic acid is then added, so that the two liquids remain in distinct layers, and at their plane of contact a red or violet ring forms should glycogen be present. In the event of extraction of the glycogen with water proving inadequate, a solution of caustic potash containing an amount of KOH equal to 3 per cent. of the weight of the flesh must be substituted.

The reaction is said to be characteristic, as it is not yielded by the flesh of other domestic animals.

B. B.

THE ANALYST.

FEBRUARY, 1894.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE annual general meeting of the members of the Society was held at Burlington House on Wednesday, January 24. The President, Sir Charles A. Cameron, occupied the chair.

The following gentlemen were proposed as members : James Edmunds, M.D., 29, Dover Street, Piccadilly ; Frederick Dunn, Flinder's Lane, West, Melbourne ; Arthur R. Ling, 45, Lambton Road, Cottenham Park, Wimbledon. The following gentlemen were duly elected : as honorary member, Dr. W. A. Tilden, Mason's College, Birmingham ; members, Dr. J. Duncan, St. Petersburg ; Percy Andrews Ellis Richards, A.I.C., Charing Cross Hospital, W.C. ; and Robert Walter Oddy, F.I.C., Waterhouse, Toad Lane, Rochdale.

The accounts for the year, which had been audited by Messrs. Chattaway and Kitto, were presented by the treasurer, Mr. E. W. Voelcker. They showed that the Society was in a more flourishing condition than at any previous period of its existence.

In presenting the report, Mr. Voelcker referred briefly to the death of the late honorary treasurer, Professor C. W. Heaton, and said that all who were personally acquainted with him had felt his loss keenly, for in him the Society had not only lost a valuable officer, but a very good friend. Mr. Heaton had been ill for a very considerable length of time, and a great deal of his work had latterly been done by Mr. Nimmo, to whom the thanks of the Society were due for the excellent way in which he had kept the accounts, and also for much valuable assistance which he had given him (Mr. Voelcker) in taking on the duties of the office of treasurer.

Mr. James Baynes moved, and Mr. E. W. J. Jones seconded : 'That the report and accounts as presented be adopted.'—This was carried unanimously.

The President moved a vote of thanks to the Chemical Society for allowing the Society the use of the convenient and well-situated room which was placed at their disposal for holding meetings ; and that this resolution be communicated to them by the honorary secretaries.

The officers for the ensuing year, as proposed by the Council, were elected.

The President then read his annual address.

Mr. Hehner proposed a cordial vote of thanks to the President for his address, which was seconded by Mr. Allen, and carried unanimously.

The President thanked the meeting for the cordial manner in which they had passed the resolution.

The members and their friends adjourned to the Criterion Restaurant, where the annual dinner took place, the President being in the chair.

THE VINEGAR QUESTION.

(Discussion held at the Ordinary Meeting, Nov. 1st, 1893, continued from page 15.)

WITH reference to the strength of vinegar, I have had several samples lately which were unreasonably dilute, one containing as little as 2.28 per cent. of acetic acid. This was taken under the Sale of Food and Drugs Act, and there is no doubt in this case it had been watered by the retailer, who was fined. The question of the limit of strength to be permitted in vinegar has quite recently acquired a new and very special interest in consequence of the action taken by a member of the Society, Mr. Estcourt, the analyst for Manchester. In Manchester there have been some prosecutions for selling watered vinegar, on Mr. Estcourt's certificate, and that gentleman regarded all vinegar as watered which contained less than 4.6 per cent. of acetic acid. There is no legalized limit of strength for vinegar, as there is now for spirits, except for pharmaceutical purposes, and the question arises whether a vinegar manufacturer has a right to sell a vinegar of any strength that he chooses. My view is that he has not. In 1874 this Society adopted "3 per cent. of real acetic acid," as the minimum limit of strength for vinegar. That is the expression used in the definitions of the Society of Public Analysts. The question arises as to what was intended by "real acetic acid" at the time, and my impression is that the term was really intended to signify acetic anhydride, $C_4H_6O_3$, which was at that time not unfrequently called "real acetic acid." But whatever was intended, the 3 per cent. limit has been generally regarded, both by public analysts and manufacturers, as referring to the compound, $C_2H_4O_2$, which is the formula of glacial acetic acid. But at any rate, whichever was the meaning to be attached to the term "real acetic acid" employed in the Society's definitions, Mr. Estcourt gave evidence to the effect that no genuine vinegar was sold in *Lancashire* with less than 4.3 per cent. of acetic acid, $C_2H_4O_2$, and on that ground he condemned a sample as containing 80 per cent. of water because it had only 3.6 per cent. (?) of acetic acid in it. Since, I have received from him the following letter, which he desires me to read to the meeting:

"In our Manchester cases we avoided any complication by asking for 'malt vinegar,' which cannot be brewed from malt and grain at so low a strength as 3 per cent. hydrated acetic acid. The manager of Messrs. Grimble and Co. admitted honestly that the vinegar they brewed was watered. This is all I certified. Of course, you will recollect the limits suggested in 1874-75 have not been adopted in their entirety, and especially is this the case with milk, where 2.5 of fat was suggested as sufficient."

It appears from this that Mr. Estcourt holds that a vinegar manufacturer has no right to dilute his vinegar with water. Whatever strength it is originally brewed it is to be kept at. He appears under the impression that the text-books are correct in stating that vinegar brewers pitch their wort according to the strength of vinegar they want to make. But in practice vinegar is always brewed of full strength, and subsequently diluted to the required strength, which is regulated by the price. I consider the maker is within his right in so doing, just as the distiller and spirit merchant have a right to dilute the spirit below the strength at which it is originally

produced. It would be absurd to employ the plant in brewing a vinegar of half strength. The different strengths of vinegar are usually distinguished by numbers, which numbers are generally regarded as representing the number of grains of carbonate of sodium neutralized by an ounce of vinegar; though of late years—in addition to the numbers 24, 22, 20, 18, and 16—"diamond," "double diamond" and "V" have been added. I believe no vinegar is sent out by the manufacturer containing less than 3·2 per cent. of acetic acid, $C_2H_4O_2$, and if it does happen it is only through carelessness. If we were to raise the present limit of strength from 3·0 to 3·5 per cent. of acetic acid, $C_2H_4O_2$, we should interfere with the sale of double-diamond vinegar, and possibly with the "diamond" of some makers, but not with No. 16 or the higher qualities. I have consulted several vinegar makers who say that the action which I now propose we should take will only mean that the cheaper vinegars will be cut out, and if the Society of Public Analysts decide that the vinegar limit should be raised to 3·8, or even 4 per cent., they are perfectly willing to accept it, provided they have due notice of the change. But, until the change is deliberately made, I think we should adhere to the 3 per cent. limit in the sense in which it has been generally understood by public analysts and respected and acted on by manufacturers.

Mr. John Pattinson, through the secretary, made the following communication :

"I beg to urge strongly that public analysts should take the position that vinegar which is bought and sold as malt vinegar, should, as its name implies, be made from malt and from malt only. Genuine malt vinegar has a special flavour and aroma of its own, which vinegar made from other sources does not possess; and the public ought to be able to obtain it when it is asked for. If it be once conceded that malt vinegar may be made from a mixture of malted and unmalted grain, it is difficult to see on what principle a vinegar made chiefly from rice, maize, or glucose, to which a few grains of malt have been added, may not also pass muster as malt vinegar, for this also could be represented as being made from malted and unmalted grain.

"Genuine malt vinegar can now be had in the market, and there are more than one firm of makers of it; but if public analysts are willing to pass vinegars made from malted and unmalted grain as malt vinegars, the public may bid farewell to all hope of ever being able to obtain genuine malt vinegar in the future, for it will be beaten out of the market by these cheaper and inferior so-called malt vinegars.

"Chemists have the means of proving fairly conclusively by analysis whether or not a vinegar has been produced from malt and unmalted barley, and they are able to distinguish such vinegar from one made largely from rice, maize, glucose, or sugar. It may be they cannot at present distinguish between vinegar made from malted barley and that made from a mixture of malted and unmalted barley, but this is not a valid reason for admitting that unmalted barley may be legitimately used in making malt vinegar. Moreover, a time may come when chemists will be able by analysis to show that unmalted barley has been used; it would therefore be unwise to admit now that unmalted barley may be legitimately used.

"I see no reason why vinegars made chiefly from unmalted barley, maize, rice, or other grain, or from sugar or molasses, should not be sold as "vinegar," or as barley vinegar, rice vinegar, grain vinegar, sugar vinegar, or by any other distinguishing name, but they should not be sold as malt vinegar.

"I am strongly of opinion that public analysts should define malt vinegar as 'an infusion of malted barley, which has undergone alcoholic and acetic fermentation.'"

Dr. W. Morgan desired to make a few observations on malt. In South Wales a large amount of malt was made by so-called maltsters. He thought that no one would for a moment doubt but that malt was a term applied to a mixture of malt and barley only. The term maltster, as he understood it, was applied to a man who produced malt from barley, and from barley only. South Wales was notorious for the distillation of pyroligneous acid from wood, and he knew, of his own personal knowledge, that what was sent out as rectified pyroligneous acid obtained by redistillation was called in the trade pyroligneous acid. There could not possibly be a trace of tarry matter in it.

Mr. A. H. Allen wished to know if he was to understand that what was known in South Wales as pyroligneous acid was a product obtained by neutralizing the acid distillate from wood with lime or soda, treating the salt obtained with sulphuric acid and re-distilling? According to his information an acid recovered from a soda or lime salt was known in the trade as "acetic acid," and distinguished from "pyroligneous acid."

Dr. W. Morgan, in reply, said that acetic acid and pyroligneous acid were synonymous terms, just in the same way that spelter and zinc were synonymous. Examination for tarry matters for the purpose of proving the presence of pyroligneous acid in a vinegar was of no value whatever. Pyroligneous acid was an unfortunate term to use, because it might convey to the mind of the uninitiated that it contained tarry matters, and all sorts of other abominations. Since his attention had been called to the matter, he was very careful in saying that it was acid obtained from wood, or other material, and not from malt. A barrister informed him that he would contend that if malt vinegar was made from a mixture of 10 per cent. of malt, and the rest starch, it was still a malt vinegar, and that it was not necessary that the vinegar should be made wholly from malt in order that it should be called malt vinegar.

Mr. G. Embrey believed that the point upon which it was really desired to come to an agreement was, that the members of the Society should agree upon what vinegar was, and what malt vinegar was, and what "faked" vinegar was. He did not think that the matter was so difficult as Mr. Allen seemed to imagine. There could be no doubt that malt was malted barley, and nothing else. No malt-vinegar brewer used malt entirely, but used 10 per cent. of malt and 90 per cent. of barley, and he malted the barley from the 10 per cent. of malt. That seemed to him the method adopted by the really honest malt-vinegar brewer. When it came to the question of what was vinegar, there could be no doubt that Mr. Hehner's definition given at Birmingham, that it was "a vegetable juice which had undergone alcoholic and acetic fermentation," was the correct one. With regard to pyroligneous acid, he did not think there ought to be any difficulty in saying that that was not vinegar at all. Dr. Morgan was quite right in saying that in South Wales the name pyroligneous acid was given to the purest acid, even if it had been derived by re-distillation or from an acetate. Public analysts ought to agree among themselves, as they had done in

former times, as to what vinegar was: the Birmingham case had shown what vinegar was not. It was also agreed that a mixture of acetic acid and burnt sugar was not vinegar. To let a manufacturer take rice, subject it to the action of sulphuric acid, and obtain an impure acid from that, and let him call it malt vinegar, seemed to him to be going a little too far.

Dr. Bernard Dyer thought that the difficulty was not so much in defining, but in finding out whether an article came under the definition. It might interest some to know that the trade in phosphate of potash was said to have increased since the vinegar agitation began; and he knew that people who sold acetic acid also dealt in phosphate of potash.

Mr. Cassal, having made some remarks with respect to certain legal and analytical aspects of the question, said that he would like to have a clear statement as to what was the position of the public analyst when he had to certify upon samples brought to him under the name of "vinegar." With regard to the distinctions between malt vinegar, alcohol vinegar, sugar vinegar, and so forth, it was highly desirable to arrive at an understanding as to dealing with mixtures of these preparations. It was of the utmost importance they should arrive at clear definitions of the articles which were submitted to them as public analysts, and he ventured to suggest that, before public analysts certified in regard to new forms of adulteration, there should have been a discussion in the Society, and a general agreement arrived at as to what ought to be done. Otherwise there would be regrettable differences of opinion in Courts of Law, and the profession as well as the public would suffer.

Mr. Hehner, in summing up the discussion, said that, taken together, the statements made by Dr. Dyer and by Mr. Allen were very remarkable, the former informing them that manufacturers in some cases were now adding phosphate of potassium to their products, in order that they might pass the analytical tests; the latter, on the contrary, that other manufacturers were abstracting phosphoric acid, in order to increase the keeping qualities of the vinegar. These statements appeared to show how little reliance could be placed upon the arguments advanced on the part of the makers. He (Mr. Hehner) feared that behind the makers stood unscrupulous chemists, who lent themselves to the screening of adulteration. It was also remarkable that no difficulty had been found in laying down the limits of composition, including phosphates, in respect to another article of infinitely greater importance than vinegar—namely, wine. Yet he (the speaker) had heard absolutely no suggestion made that wine was largely "faked" with a view of circumventing the analyst. He was convinced that the present tricks adopted by one portion of the vinegar trade were but passing events, and that the trade would not only soon find its level again, but find itself all the better and purer for the agitation, upon which so much comment had been made.

Mr. Embrey, he thought, had emphasized the main point in the discussion when he said that the chief requirement was a decision as to what malt-vinegar was. It had been decided, and fairly conclusively so, that wood-acid was not vinegar. There was also an agreement of opinion that vinegar, without a qualifying adjunctive, might

be made from many sources, provided it was a real product of alcoholic and acetic fermentation; but there was no manner of agreement as to the nature of malt-vinegar. In the Birmingham case, at its first (Police Court) hearing, the chemists giving evidence for the prosecution thought that all vinegar should be malt-vinegar, unless expressly acknowledged to be made from other sources. But on closer consideration it was seen that this was an untenable position, and one which would much interfere with legitimate vinegar manufacture; and it was then agreed that vinegar should be defined as a vegetable juice or infusion which had undergone alcoholic and acetic fermentation. Mr. Allen had shown to the members of the Society the many difficulties involved by the adoption of that definition. The next question which was sure to come prominently to the front was, in his (Mr. Hehner's) opinion, the following, What ought malt-vinegar to be made from? Mr. Pattinson took the view that malt-vinegar should be strictly what the name implies, made from malt and water only. This was the real logical and common-sense view of the matter, but as analytical knowledge of the present time did not enable public analysts to distinguish between vinegars made from malt only, and those made from malted and unmalted barley, it appeared to him to be useless to insist upon a definition which could not be controlled or enforced; and he was of opinion that it was better to adopt, for the present at least, the definition that malt-vinegar was the product of the alcoholic and acetic fermentation of an infusion of malted and unmalted barley. He could not concede for a moment that starch hydrolyzed by sulphuric acid might be substituted for the inversion by diastase without altering the character of the product. Only the widest chemical outlines of the chemistry of fermentation were at present understood, and we were in ignorance of the causes which determine the physiological effects of the products of fermentation. The chemist was far too apt to jump to the conclusion that, because the main products of a process had been ascertained, therefore we knew and understood the process. The argument had often been employed in connection with this matter, that, inasmuch as starch yielded on inversion by any process sugar, which in turn yielded alcohol, from which the acetic acid of the vinegar was derived, therefore one source of acetic acid was as good as another. But though pure alcohol might be the same from whatever source it came, it was a fact beyond dispute that the products of fermentation of grape-juice were wholesome, and not liable to produce unpleasant physiological effects beyond those attached to pure alcohol, while the products of fermentation of, say, potato-starch sugar were physiologically most objectionable and injurious.

For the sake of ordinary commercial honesty, and in order to uphold the spirit and intention of the Sale of Food and Drugs Act, analysts should, in his opinion, insist that an article called by a specific and definite name should be made of the specific and definite article which gave it its appellation. No one could object to sugar-vinegar being honestly sold under that name. He went further, and thought that if a manufacturer called dilute wood-acid "wood-vinegar" he was acting straightforwardly and did not deceive purchasers. Similarly, it might be expected that if a maker sold vinegar under the name of malt-vinegar, the public had a right to insist that the article should be really made from malt, and that without the slightest equivocation.

As to the proportion of acetic acid in vinegar, he would like analysts to be careful not to come rashly to conclusions. He had lately met with a sample of vinegar which had within a very short time deteriorated in the cask by growth of fungus, the acetic acid falling from upwards of 4 per cent. to as little as 2.9, watering being excluded from the circumstance that all other constituents of the sample had remained unaltered. When a barrel was nearly empty and the surface of the fluid exposed largely to the air, the contents often rapidly lost in strength. Of course, the vendors should be obliged to sell vinegar of fair strength, and it could be no excuse to plead that the article had altered from natural causes, but the lowest strength did not necessarily imply intentional and fraudulent watering.

He believed that in most cases analysis enabled the chemist to obtain a very good idea of the materials employed in the manufacture of the sample. The presence of aldehyde and alcohol, causing an abundant iodoform reaction in the distillate from the neutralized sample, was fairly conclusive evidence of fermentation, and the sample was a real vinegar. Vinegar made from sugar contained hardly any albuminoids, while that from malt contained about 0.7 per cent. Vinegar prepared by acid inversion of rice and other starch always contained a high ash with abundance of sulphates. The ash of a cane-sugar vinegar was readily fusible, even over a moderate argand flame; that of a malt or a glucose vinegar did not readily fuse. Sugar-vinegar yielded an ash composed mainly of potash salts, as raw cane-sugar was employed, and never refined sugar. For that reason he thought that the estimation of potash with a view to prove the presence of grain vinegar was useless, both grain and raw sugar containing much potash.

Mr. Allen had referred to his (Mr. Hehner's) mode of calculation of the original total solids, and had stated that, as it did not take any account of the loss involved in the process of manufacture, it might be improved upon. No doubt it might be improved, but when losses were taken into consideration, the analyst would at once be required to give his reasons for any assumption he might make. And the loss varied very much in different factories. Besides, the method of calculating the original total solids adopted by him (Mr. Hehner) was the most favourable that could be adopted, all errors being to the benefit of the manufacturer.

It had not been altogether fortunate that Dr. Hill had been successful in the first case brought against wood vinegar. There was no chemical evidence that the sample consisted of wood-acid, although the makers freely acknowledged that Dr. Hill's conclusions were quite correct. But other public analysts following Dr. Hill had often not been so fortunate in their conclusions, for, as they now knew, it by no means followed that because a vinegar was not malt-vinegar it must necessarily contain wood-acid. It was advisable to be exceedingly cautious in the expression of an opinion, and all the circumstances of the case should be taken into consideration. Supposing an analyst examined a vinegar which contained 4 per cent. of total solids and 1 per cent. of ash (an actual case), but which was almost devoid of phosphoric acid, it would be rash and injudicious for him to say that that sample contained wood-acid in proportion to the deficiency of phosphoric acid—say, 75 per cent.—because the 4 per cent. of total solids would not be derived from the wood-acid, which

constituted three-fourths of the article, but from the one-fourth of real vinegar, and this, therefore, would have had to contain 16 per cent. of total solids, which would be absurd. The same argument applied to the ash, brought that of the real vinegar taken to 4 per cent., and this was equally impossible. As a matter of fact, the sample in question was composed of a mixture of a glucose and a sugar vinegar, and was quite devoid of wood-acid.

He wished, lastly, to say a few words as to the legitimacy of removing any substance which was an essential constituent of any commercial article, and without which it was not the real commercial article. He wished to protest against the removal of phosphoric acid or of albuminoids from vinegar, and thought that the manufacturer had absolutely no right to effect such a removal. Would anyone tolerate the removal of phosphoric acid or of tartaric acid from wine? The removal was prejudicial to the purchaser, and its sole object was to enable the maker to obviate the proper process of ageing which the article ought to be subjected to, and to hasten the sale of an otherwise immature and unsaleable article. Here, again, he was of opinion that the long-recognised and legitimate modes of manufacture should be adhered to, and the introduction of chemical meddling with food materials resisted. It should not be left to the discretion of the manufacturer of articles of food to say which constituents were valuable and essential and which were not, and in no case should such removal be effected without due notice to the purchaser.

Mr. Allen, in reply, said that he entirely disagreed with Mr. Hehner that a vinegar manufacturer was not at liberty to remove things prejudicial to vinegar. It was, in his opinion, the manufacturer's business to make good vinegar, and so long as he sold it for what it was, he was at perfect liberty to remove any objectionable constituents which impaired its keeping properties. The reason why malt vinegar brewers desired to remove the albuminoids was that they rendered the vinegar particularly apt to undergo secondary decomposition. He believed the decomposition of vinegar, with consequent change of strength, would always be accompanied with a very visible formation of mould, and that the vinegar would become turbid. He usually regarded the presence of potash as distinguishing true fermentation vinegar from the outside products, and he quite agreed with Mr. Hehner that its value would be diminished when trying to discriminate between different kinds of fermentation vinegar. He had been recently asked in Court if he knew a book called "Commercial Organic Analysis," in which there was a statement made that "malt vinegars have a high density (1021 to 1025), and yielded 5 to 6 of extract containing a notable proportion of phosphates." He believed that this statement was true at the time it was written, some ten years ago: the fact being, that vinegar-brewers conducted their fermentation better at the present time—they pushed it further, leaving less sugar in, and, at the same time, there were less albuminoids than there used to be formerly. Hence vinegar kept much better, as now manufactured, than it did formerly, and there was no excuse for the addition of sulphuric acid as a preservative, as was formerly permitted by the Excise.

Mr. Embrey suggested that the matter should be referred to the Council for report, and that the report should be considered by the Society at a special meeting.

The following letter has been subsequently received from Mr. E. W. T. Jones :

" I consider it would be a calamity for the Society to admit a deviation from the strictly scientific formula, viz., a *product initiated by malt only* ; raw grain may be used with it, in the mash-tun, because the utilization of its starch is absolutely restricted to the action of the malt, and therefore the constituents of the wort may be said to be strictly malt products. I would not admit the products of starch hydrolyzed by sulphuric acid or in other ways than by diastase. This gives a glucose vinegar.

" I know some of my friends will say, Can we analytically differentiate these products and mixtures of them when made into vinegar, and if we cannot fully do this, what is the use of making any fuss about it? But I reply that, as purists, we must adhere to a principle, and what may not be an easy analytical problem to-day, may by work and progress become one to-morrow. As public analysts, is not this our experience over and over again?

" A wort made by the acid degradation of starch is not the same as one made by diastatic hydrolysis with the latter; under the most favourable circumstances the transformation does not go practically beyond four-fifths maltose and one-fifth dextrin, and much of this latter remains in the finished vinegar and gives it 'body,' but the acid conversion, as *carried out*, produces dextrose, gallisin, etc. : little or no dextrin is left to give 'body' to the vinegar. Oh, but some of my glucose friends say, what is the value of the dextrin or 'body'? Vinegar without it is preferable, and our product for several reasons will keep better. I do not care if all and more than this is true—as public analysts we have nothing to do with such a question—if malt vinegar is asked for it must be supplied, and the public must decide by their demand what it is they require.

" I may mention that genuine malt vinegar, agreeing with the formula I give, is being made on a very large scale, so that I am not insisting on anything impracticable. I must also admit that most of the vinegar now sold as 'malt' is mixed with the glucose product, but in time public analysts must remedy this."

VINEGAR.

BY EDWARD COLLENS.

(*Read at the Meeting, December 6th, 1893.*)

Messrs. Alleh and Moor, in their paper on vinegar, published in your issue of October, 1893 (vol. xviii., p. 240), have fallen into certain errors in their treatment of the history of the subject, which I will endeavour to correct.

In the first place, one is led to believe that the use of raw grain is a very recent departure, consequent on the example set the vinegar-maker by the brewer. This is such a complete inversion of fact that a short account of the doings of the former during the last hundred years will be the speediest reply.

Originally molasses was the only raw material used in the manufacture of English commercial vinegar. The West Indian planter conducted his operations on a most wasteful plan, and the distiller and vinegar-maker profited by it; hence the old vinegar houses are established in the ports which were then the centres of the West Indian trade, or in places within easy touch of these, *e.g.*, London, Bristol, Stourport; and to this day a survival of the molasses-vinegar trade remains, for the malt vinegar-maker must colour his paler article to make it resemble the older vinegar ere the consumer will purchase it.

But with increasing chemical knowledge, good molasses ceased to be a drug in the market, and the vinegar-maker and his friend the distiller—for in their earliest stages the two trades are, of course, identical—availed themselves of the fact that a mixture of malt and raw corn when suitably treated gave them all they wanted.

This product was advertised on the show-cards of the day as 'Patent Malt Vinegar,' but the word 'patent,' I am inclined to think, if it had any justification at all, referred to the apparatus used in its manufacture, the acetifier, then patented by one Ham, of Bristol, and which was for all practical purposes the same as is used to-day.

This process held its own without much variation until recent years, when the great cheapening of sugar, consequent on the beetroot industry, assisted by the abolition of the sugar-duty in 1874, again made the use of that article possible.

The second statement I am inclined to challenge is this: "There is undoubtedly a popular preference for, or prejudice in favour of, malt vinegar." My own experience is that so long as the consumer gets *vinegar*—*i.e.*, "a product of the consecutive alcoholic and acetic fermentations"—it is all he wants.

I recently asked a dealer of large experience, extending over thirty years, if *malt vinegar* was ever asked for. "No!" said he, "by no one but the inspector, and he is so conscious of asking for something unusual, that he demands *vinegar* and gets the admission that it is *malt vinegar* in course of conversation." And it would be strange if he had, for malt is not added to the mash, as is asserted by the authors of the paper, for the sake of giving a flavour. The one sole object for which it is used is the hydrolysis of the starch: it is a reagent, and nothing more; and no physical or chemical test whatever can show where malt is used to the exclusion of unmalted grain, or where it is wholly absent. The vinegars are identical; therefore to talk of apple or turnip pulp flavoured with raspberry, as an analogous case, is entirely erroneous.

If the turnip could be so treated as to cause it to assume the chemical and physical characteristics of raspberry, the simile would be borne out, but in that case why should it not be called raspberry jam?

I entirely agree with the authors of the paper in their desire to see the designation "malt vinegar" abolished; it was so, practically, until within the last six months, but the wording of the summonses in recent prosecutions, and the action of the inspectors in demanding malt vinegar, has brought it back again.

My only other criticism concerns the statement that there is an import duty on vinegar. There was a charge of threepence per gallon, but it was repealed in 1880,

probably in consequence of the fact that by the abolition of the malt tax the home-manufacturer no longer contributed to the Excise revenue.

I would like to appeal to those of you who are public analysts to consider the absurdly unequal way in which the phosphoric acid standard affects vinegar-makers. Let us assume two manufacturers working on the same lines; they shall both use rice as their raw grain, as legitimate a source of starch as rye, barley, oats, or maize, but one shall be a worshipper of the good old rule of thumb, and the other a believer in the doctrine that to waste material in manufacturing operations is to sin against science. The first uses more than sufficient malt for the supply of the diastase he requires; he purchases a dirty rice with plenty of adherent skin and husk; he gets bad fermentations with consequent low percentage of alcohol, and that is further wasted by unskilful acetification. The other uses his malt with more judgment; he hardens his brewing water for very obvious reasons; he buys perfectly clean rice; he closely watches his attenuations; and if results in this and the acetifier stage do not closely accord with the theoretical quantity, he is not satisfied until he has found a reason and a remedy. But whilst the vinegar of the first maker will show abundant phosphates, that of the second will only indicate some 0.2 or 0.3 of P_2O_5 in "total solids" as calculated, and he must either condescend to a trick in order to make his vinegar square with the ideas of a self-constituted and only partially informed tribunal; or he must take the risk of having to dance attendance at half the Petty Sessional Courts of the kingdom.

DISCUSSION.

Mr. Allen was glad that the paper which he and Mr. Moor read on this subject should have elicited this communication from Mr. Collens. He had not the pleasure of knowing that gentleman, but from the style of his arguments he assumed that he had a close connection with the vinegar trade. They (Messrs. Allen and Moor) were indebted to him for pointing out errors from an historical point of view. Their information was not faultless in these respects, and he was glad that anyone should have placed these matters correctly before them. On the other hand, he thought Mr. Collens rather missed the point raised on the last occasion where he (Mr. Allen) tried to explain the position which was occupied with reference to these different materials, and how far the articles should be known as "vinegar" or "malt vinegar." There were manufacturers who gave the term "malt vinegar" to an article which was not brewed from malted and unmalted grain. Manufacturers were anxious to sell products of mixed origin as malt vinegar, and they had put considerable pressure on himself and other chemists to say they regarded such as malt vinegar. He expressed his own opinion on the subject at the last meeting, and he was sorry they had not got any definite expression of opinion from the Society, for it was really a burning question. Yesterday he attended the Petty Sessions at Keighley in support of a prosecution for selling as "malt vinegar" an article which he certified to contain the parts as under:

Malt vinegar	50 parts
Vinegar from sources other than malt (probably sugar)	50	..					
	<hr/>						100

As an "observation," he explained that by malt vinegar he understood a vinegar

brewed from a mixture of malted and unmalted grain. The manufacturers, Messrs. Grimble and Co., Limited, admitted the accuracy of the certificate, and stated that the vinegar was brewed from a mixture of malted and unmalted grain with sugar, but they contended that such an article had a right to be considered as genuine malt vinegar. In proof of this, they stated that they annually submitted samples of their malt vinegar to the Admiralty, which samples were analyzed at Somerset House; and when they failed to obtain the contract they inquired the reason, and had never been told that their product was rejected as not being genuine malt vinegar. In consequence of this indirect testimonial from Somerset House, and for want of an authoritative definition of malt vinegar, the magistrates dismissed the case. It was perfectly evident from recent cases that Somerset House were prepared to pass as genuine malt vinegar anything that was not distilled acetic acid in all its nakedness, and even that they would call "an artificial vinegar of commerce." Mr. Collens said if they could make a factitious raspberry jam so skilfully that a chemist could not distinguish it from true raspberry jam, it would have every right to be called raspberry jam. This was a most improper and immoral position. As chemists they knew their powers were limited, and it was preposterous to say that an imitation article had a right to be sold in place of the genuine because, at the present time, analysts could not distinguish the difference. If a customer asked for malt vinegar or raspberry jam he should be served with it. Let them take the case of a person who was a purchaser. He sees a bottle labelled "Pure malt vinegar," "Try so-and-so's pure malt vinegar," "Beware of imitations." Then the vinegar manufacturers come forward and say: "When we advertise malt vinegar we do not expect you to believe us. We don't mean malt vinegar when we so describe it; the people don't want malt vinegar; our vinegar is much better than malt vinegar, and keeps better." It seemed to him (Mr. Allen) an improper and illogical position to take up.

The Chairman (Mr. Hehner) said that whether the history of Mr. Collens was correct or not had not much to do with the question, but he thought that gentleman looked at the subject solely from the manufacturer's point of view. He had asked, Can an analyst discover whether it was malt vinegar or not? and he (the Chairman) thought that Mr. Collens gave the proper keynote to his paper when he gave the illustration about raspberry jam, and argued that, because the chemist might not be able to distinguish adulteration, surely that there should be the right to sell it as raspberry jam. If that were so, where would commercial honesty be? He was entitled to obtain that for which he asked; whether there was sufficient reason, it was not the chemist's or vendor's business to gauge. He asked for a thing, and the principle on which the whole Food Act was constructed was that he should have that for which he asked. Supposing, as they all knew to be the fact, that for one hundred years or thereabouts pure brandy had not been used in this country or not to any large extent, and that a very large proportion of brandy had been made from silent spirit, and that millions of gallons had been sold under the Food and Drugs Act, surely that did not give sanction to it; and if chemists could distinguish between real wine-brandy and the artificial mixture, would they not do so? Mr. Collens had entirely disregarded the principle of commercial honesty on which trade ought to be conducted.

AN IMPROVEMENT IN RICHMOND'S MILK-SCALE.

BY CHARLES E. CASSAL AND B. HENRY GERRANS.

(Read at the Meeting December 6th, 1893.)

We desire to show the members of the Society an improvement in the well-known piece of apparatus which we owe to our friend Mr. Richmond. Those who have been accustomed to use the slide-scale of Mr. Richmond to any great extent will no doubt have found that, at times, if not repeatedly, one loses the first number when one is looking for the second, and that an appreciable amount of time is thus lost in working with the scale. The main improvements consist in a movable index which marks the percentage of total solids on the scale, and in another movable index which can be placed on the number indicating the specific gravity. By putting the points of the adjusted needles opposite each other, one gets the desired results with far less mental strain. It is unnecessary for me to enlarge upon the advantages we have found from these additions. The improved scale has been made by Messrs. Baird and Tatlock. The only objection to the one now shown is that the needle for the total solid scale is rather long; it should be shorter and finer, to enable the graduations to be seen more easily. In addition, we have introduced a couple of knobs at each end of the movable scale, to enable the slide to be moved more readily. Another suggestion which, I think, is of importance—but which Messrs. Baird and Tatlock inform me cannot be carried out without considerable expense at present—is the further subdivision of the total solid scale and of the fat scale. While the formula on which the scale is based is perhaps not absolute, we think Mr. Richmond will agree with us that there is no reason why the subdivision should not be carried further. We mention this so that when these scales are re-manufactured a further subdivision should be introduced. It will be found that the improvements now shown render the use of the scale practically automatic. It may be mentioned that we do not necessarily bind ourselves to the particular form of the apparatus now shown. In this form, Messrs. Baird and Tatlock were best able to carry out what we required with the minimum of expense. We have used another form of the apparatus for a long time, in which the indicator is only applied to the total solid figures; the way in which the piece of metal clasps the scale is perhaps a little more satisfactory in this form.

Mr. Richmond said the improvement was ingenious, but, as far as he was personally concerned, he found it to be a matter of habit and training to find the two numbers almost instantaneously. If when finding one he kept his eye on it until he came to the other, he found it was done quickly. Personally, he did not feel to want the aid of a pointer to help him to come to that idea, but he could quite understand that to many men it would be an advantage. Some five years ago exactly the same idea was suggested to him, but he had always thought that it was not necessary. As Mr. Cassal, however, had done it, and produced a neat-looking instrument, he hoped those people who found it convenient would take his (Mr. Cassal's) view.

Mr. Cassal, in reply to a question from the Chairman, said that the cost of

the improved scale was 7s. 6d. Messrs. Baird and Tatlock now sold the original scale at 5s.; and with the additions now shown they proposed to charge an extra 2s. 6d. It was perhaps necessary to say that Mr. Gerrans and himself had not patented the improvement with a view to derive any pecuniary advantage from its sale. He was sure that those analysts who had been in the habit of using this scale every day and very extensively would agree that it was easier to have the figures marked out by what amounted to an automatic process, and that this was better than a process which involved more time and fatigue. Mr. Gerrans and himself had found these improvements extremely useful; they saved a very considerable amount of time, involved far less mental strain in carrying out work—when, say, twenty or thirty milk analyses had to be dealt with—and the operations performed with the scale were more certain and accurate. Even people possessed of the remarkable powers described might make mistakes. The improved scale would be found to afford a guarantee against error.

The Chairman thought the improvement would be very useful to many. He himself often had to look twice for the figures. It would help to make this scale a still more useful article, by making it still more easily used. He thought the milk-scale of Mr. Richmond had hardly had the recognition which it deserved. When visiting other laboratories and making a request for a milk-scale, he had often found it absent, although he (Mr. Hehner) had found it indispensable.

AN ADJUNCT TO THE MILK-SCALE.

By H. DROOP RICHMOND.

Messrs. Cassal and Gerrans lately exhibited to the Society a milk-scale with pointers to mark the specific gravity and total solids from which it is desired to calculate the fat.

I mentioned in the discussion that Mr. A. E. Johnson had previously suggested the idea to me, and that since then other gentlemen had also mentioned it; personally—perhaps from continued usage of the milk-scale—I had not felt the least difficulty in adjusting the slide without such aid. I suggested to Mr. Johnson that, by turning the slide round so that the arrow pointed to the total solids, he would find the use of a pointer unnecessary, and have frequently used this way of working the instrument, which has, however, the disadvantage that the figures on the specific gravity slide are inverted. Since seeing the arrangement of Messrs. Cassal and Gerrans, I have given the use of a pointer a serious trial, with the result that both I and my assistants find it a relief to the mental strain involved in calculating many results.

The modification of Messrs. Cassal and Gerrans is unnecessarily complicated; the use of two pointers is not needed, and increases the trouble of using it, and in the instruments exhibited they were liable to shift. The annexed figure shows a slide made from a piece of sheet-brass, bent so as to encircle the scale, the hinder

portion acting as a spring. The line is adjusted to the total solids; the specific gravity is then brought even with the line, and the fat is read off by the arrow.

In the figure decimal scales are placed by the side of the line which shows the total solids and the arrow which shows the fat, and by these results can be read to .01 per cent. I do not recommend this addition, as the divisions are so small as to fatigue the eye, and the accuracy of the graduation must be to at least $\frac{1}{200}$ of an inch, which will greatly increase the cost of the instrument; they are placed in the figure simply to show that readings to .01 per cent. could be obtained if necessary.

WATER ANALYSIS—THE INTERPRETATION OF RESULTS—*Concluded.*

By F. WALLIS STODDART.

Reprinted from the "Practitioner."

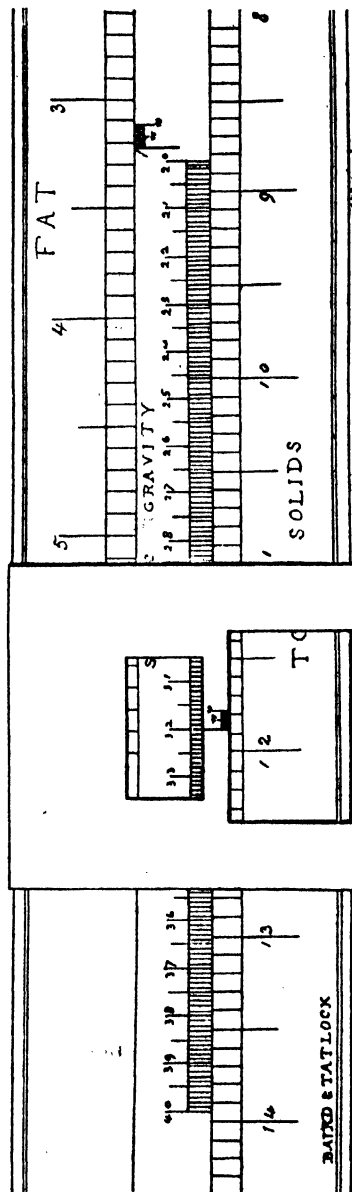
GENERAL CONCLUSIONS.

THE preceding considerations, then, appear to me clearly to prove—

(1) That natural waters can at most obtain from one-tenth to two-tenths of grain of nitrogen as nitrates per gallon from sources other than animal matter.

(2) That practically the whole of the nitrogen of sewage may be oxidized into nitric acid without materially diminishing the risk involved in drinking it.

Let us, then, state the case in this way. The one most characteristic constituent of sewage is nitrogen; in the absence of nitrogen in some form of combination it cannot be said that water is polluted with sewage. This nitrogen contained in the original excrement as a constituent of organic compounds, assumes, as it were, a series of disguises under the influence of a succession of fermentative processes, appearing at one time as ammonia, at another as nitrite, at a third as nitrate, and so on, probably becoming at no distant period the free element. All this time it may be accompanied by the specific infective matter of cholera, enteric fever, and probably other diseases.



It is the duty, then, of the water analyst to detect the nitrogen under these disguises, gathering, of course, all additional information obtainable from other determinations; and as we have no reliable proof as to when the danger, of which the detection of sewage products is the sole indication, ceases, we must regard it as still existing so long as the chemical evidence of pollution is forthcoming.

When the proportion of nitrogen, in whatever form, amounts to a grain per gallon and upwards, so that the water plainly consists largely of sewage whether oxidized or not, the danger of drinking it must be obvious to anyone who bestows a thought on the matter. For even if the view be not adopted that the specific poison may persist in the water at this stage, the presence of so large a proportion of sewage products argues close proximity to the source of pollution; whilst if it be admitted, as I think it must, that the infective character of the sewage may be unimpaired, the danger is all the more imminent. The proposal to consider a water safe so soon as the nitrogen has assumed the oxidized condition, irrespective of the quantity that may be present, is then entirely irrational, and incapable of support by any kind of argument. Indeed, there are cases on record where outbreaks of typhoid have been traced to water of this character,* and there are others where the extent of percolation was so great, as, for instance, in the well-known Lausen and Havre epidemics, that nitrification must have been completed. In the Havre case the polluted water must have percolated through no less than 45 metres of chalk before reaching the spring-head; and it is impossible to imagine that any appreciable amount of nitrogen escaped oxidation, though I have been unable to obtain any analysis of the water.

It is a more difficult matter to decide whether we should permit any evidence of this form of contamination at all in a water. It appears clear to me that, whatever class of water is under consideration, seeing that average sewage contains about 5 grains only of nitrogen per gallon, a quantity of nitrogen as nitrates exceeding 0.5 grain per gallon indicates either dangerous proximity of the well to a source of pollution, or such easy communication with it that the distance separating the two points is no guarantee of purification: hence such water should be regarded with the greatest suspicion. With smaller quantities than this it becomes desirable to take into consideration the special circumstances of each case particularly, with a view to ascertaining whether the small excess is due to a slight admixture of recent sewage, or to a nearly complete removal of the sewage products by natural filtration. In the former case the water would be in the same position as that which was lately the cause of an epidemic in Chester-le-Street,† where, owing to dilution of the specifically infected sewage, no very definite chemical evidence of pollution was obtainable; in the latter everything depends upon the confidence placed in the purifying effect of natural filtration, in which connection the above-mentioned cases are of obvious importance.

I have purposely refrained from referring more than was unavoidable to the opinion expressed by English writers on this subject, partly because their views are

* Cf. *Supplementary Report of Medical Officer of the Local Government Board*, 1874; Haines, *Journ. Franklin Inst.*, 1881, etc.

† Report by Dr. T. Eustace Hill to the Durham County Council, April, 1893.

well known, and partly because it is very undesirable to perpetuate any personal feeling, such as was at one time associated with the question in most minds. But I may be permitted to refer to what I consider to be one of the most important contributions to the whole subject of water analysis ever published, I mean the report by Professor Mallet to the American National Board of Health on the Determination of Organic Matter in Potable Water. This report contains a very detailed examination of all the leading processes, and contains amongst other conclusions this remark: "No aspect in which I have compared together the good and bad natural waters has afforded so definite a result as this," *i.e.* the presence of nitrates and nitrites.

In conclusion, I may be permitted to give the essential points of the case which induced me to pen the preceding remarks. A shallow well in millstone grit, and under the most unsatisfactory conditions possible as regards surroundings, was found to yield water largely polluted with oxidized sewage, and was accordingly condemned. Opposition being made, a sample was ordered to be sent to Somerset House, and the results of analysis obtained by the chemical staff there, practically corroborating mine, were as follows:

						Grs. per gal.
Free ammonia, '03 per mil.	=	'002
Albuminoid ammonia, '068 per mil.	=	'0048
Oxygen absorbed in three hours '037 per 100,000	=	'026
Chlorides (as sodium chloride)	=	5.9
= chlorine	=	3.58
Nitrates (as nitric acid)	=	9.4
= nitrogen...	=	2.44
Total solids	=	55.5

The opinion appended was that "although the water is unsatisfactory in some respects, and one we should not recommend, we do not feel justified, from a consideration of the results of the analysis, in giving an opinion that the use of the water for potable purposes would be dangerous to health."

Now, if there were any alternative explanation of the composition of this water still, having regard to the insanitary condition of the surroundings, I maintain one would be justified in condemning the water as dangerous to health. But when it is incontestable that the water consists very largely of the leakage from drains, the proximity of which to the well was clearly explained to the Somerset House chemists, and which, indeed, they refer to as "almost dangerous" in the earlier part of their Report, it is perfectly unintelligible how anyone accustomed to weigh evidence can escape the conviction that this well is a constant source of danger to the very numerous persons who habitually use it.

However, my present object will be attained if I succeed in inducing those who are engaged in water analysis, and who have been hitherto content to ignore nitrates altogether, to take the question into serious consideration. I am convinced the result will go far towards putting an end to the possibility of extensive outbreaks of cholera or typhoid amongst our crowded populations, now to an unsuspected extent dependent upon the defects in their own drainage systems for their water supply.

Cotton-seed Stearin and Maize-oil. F. Hart. (*Chem. Zeit.*, 1893, xvii. 1522.)—The comparatively scanty literature concerning cotton-seed stearin and maize-oil (cf. ANALYST, 1893, 191), and the considerable use of the former as an adulterant of lard, have led the author to give the analytical figures obtained by the examination of samples obtained direct from the manufacturer. Spuller's results for maize-oil are appended for the sake of comparison:

	Cotton-seed Stearin.	Hart.	Maize-oil.	Spuller.
Colour and consistency	bright yellow ; buttery	dark-brown ; fluid		—
Specific gravity	0·867 at 100°C.	0·9239 at 15°C.		—
Melting-point	30—31°C.	—		—
Melting-point of fatty acids	45·5—46·5°C.	25°C.		—
Free fatty acid calculated as oleic acid	neutral	0·75%		—
Hehner's number	96·3	95·7		94·7
Saponification number	194·6	189·5		188·1—189·2
" " of fatty acids	—	—		198·4
Iodine absorption	93·6	117·0		119·4—119·9
" " of fatty acids	—	—		125·0
Unsaponifiable matter	0·56%	1·55%		1·35%
Rise of temperature with sulphuric acid	48°C.	60·5°C.		56·0°C
Colour reaction with sul- phuric acid	dark purple red	fine dark red		—
Reaction with Bechi's re- agent	deep black	slightly darkened		—
Reaction with Wellman's reagent	dark green, fine blue with NH ₃	dark green, fine blue with NH ₃		—

From these figures it appears that, although cotton-seed stearin is well adapted for use as an adulterant of margarine and lard, yet it can be fairly easily detected by the determination of the iodine absorption, by the rise of temperature with sulphuric acid, and by Bechi's and Wellman's tests. B. B.

The Determination of Alkali in Alkaline Arsenites. G. Favrel. (*J. Pharm. Chim.*, 1893, xxviii. 301; through *Chem. Zeit.*)—Cochineal is indifferent to arsenious acid, on which account it can be used to determine the total alkali in alkaline arsenites, the examination of which resolves itself into the titration of the arsenious acid with iodine and the determination of the base in the manner mentioned above. B. B.

Detection of Saccharin in Wines, Liqueurs, or Beer. E. Spaeth. (*Zeits. angewand. Chem.*, 1893, 579.) The isolation of saccharin from wines and liqueurs, by means of a separatory funnel and immiscible solvent, is not advisable, since gelatinization of the mixture is very apt to occur, which renders the separation of

the solvent a tedious and troublesome operation. The author proposes the following method, which is somewhat similar to that used for the separation of glycerin from wines or beer. In operating with beer, unless the hop resin be previously removed, it is extracted along with the saccharin, and masks the sweet taste of the latter. For wines and liqueurs the sample is evaporated to 10 or 20 c.c. with coarse sand in a porcelain dish, and the residue, after treatment with 1 or 2 c.c. of phosphoric acid, is extracted by gently warming it on the water-bath with a mixture of equal parts of ether and petroleum ether (b.p. 60°), and breaking it up with a glass rod. The solution is filtered through purified asbestos (filter-paper takes up saccharin) and the extraction repeated until 200 to 250 c.c. of filtrate have been obtained. The solvent is next distilled off on the water-bath, and the residue freed from the last traces of it by warming and blowing in air. A few drops of a well-diluted solution of sodium carbonate are then added to dissolve the saccharin, which may here be detected by its taste, and the contents of the flask are washed into a porcelain dish and evaporated nearly to dryness. From 1 to 2 grammes of solid sodium carbonate are now added, and the mixture is gradually added in small portions to fused potassium nitrate. The melt is dissolved in water and the sulphuric acid in it determined.

For beer, the hop-bitters are first removed by precipitation with copper nitrate. 500 c.c. of the beer are mixed in a dish with a few crystals of copper nitrate, whereby the hop-bitters are thrown down as a flocculent precipitate, which, however, need not be filtered off. The liquid is evaporated to a thin syrup, a sufficient quantity of coarse sand, and a few c.c. of phosphoric acid are added. The extraction, with a mixture of ether and petroleum-ether is conducted as described for wines. So little as 0.001 per cent. of saccharin can be detected by the sweet taste of the sodium carbonate solution of the ether and petroleum ether residue. The estimation of the saccharin may be performed by fusion with potassium nitrate as described above.

When very small quantities of saccharin have to be estimated a larger quantity of beer must be used, in which case the sandy residue becomes too large and unwieldy for extraction with the solvent. In such a case the acidified sandy residue may be first extracted with warm alcohol, the alcoholic solution filtered through asbestos, evaporated, and the residue extracted as already described with the mixture of ether and petroleum ether.

A. G. B.

The Analysis of Rubber Goods. D. Holde (*Chem. Zeit.*, 1893, xvii. 1634-35).

—The author, continuing his investigations on the improvement of methods for the proximate analysis of rubber goods (*THE ANALYST*, xviii. 147), has endeavoured to ascertain whether the fact mentioned by Henriques in the latter's papers on the same subject (*THE ANALYST*, xviii. 13), that ordinary organic solvents extract an appreciable amount of matter from pure rubber, invalidates his (the author's) process for the determination of mineral and fatty oil in rubber mixtures. The solvent used by the author is ether-alcohol in the proportion of 4 to 3, and the chief difficulty apprehended was the possible necessity for repeated extraction to remove the whole of the fatty or mineral oil, as by such repetition the amount of extract from the rubber

itself would be unduly increased. Experiments were therefore made to determine whether satisfactory extraction of the oil without too great solution of the rubber could be secured. The general procedure consisted in reducing the rubber to as fine a state of division as practicable by filing it with a clean file, and extracting it on an ordinary filter-paper with known quantities of the solvent (ether-alcohol), evaporating the solution and weighing the residue. This was done with pure rubber, and with the same rubber which had been allowed to soak up known amounts of various oils. The chief results are recorded in the following table:

Kind of oil and rubber.			Percentage of oil used.		Percentage of oil found.	
White erasing rubber + rape-oil	22.1	22.1
Ditto ditto	12.9	14.1
Ditto ditto	18.5	20.2
Ditto ditto	26.8	28.4
Ditto + cotton-seed-oil	59.8	59.7
Rubber cork + rape-oil	15.7	12.9
Ditto ditto	26.2	24.2
White rubber-tube + rape-oil	26.7	23.8
White rubber-tube + cottonseed-oil	39.3	37.7
Ditto ditto	60.2	58.6

The samples of rubber yielded amounts of soluble matter, consisting of the oily constituent of rubber and free sulphur, varying from 3.7 per cent. to 7.1 per cent. The values given above for the quantity of oil found, have been corrected by the deduction of 5 per cent. from the percentage actually obtained, that being a fair mean from the results of the experiments on pure rubber. In general 60 c.c. of the solvent were used, although 30 c.c. were found sufficient in some cases. The chief cause for the discrepancies of the observed and true percentages of oil is the variation of the quantity of soluble matter yielded by the rubber itself. Greater accuracy could be attained if necessary by the determination of the free sulphur in the soluble matter, thus eliminating one of the two unknown quantities. B. B.

Kapok-Oil. R. Henriques. (*Chem. Zeit.*, 1893, xvii. 1283.)—Certain plants of the *Malvaceæ* resemble those classed under the title *Gossypium* (the true cotton-plants) in possessing a filamentous covering on their seeds. Inferiority in mechanical properties, e.g., length and strength of fibre, prevents the use of this material for the same purposes as cotton. The best known of these plants is the *Kapok* (*Eriodendron anfractosum*, Dec., *Bombax penandrum*, L.), which grows in the Dutch East Indies; its cotton is used for stuffing beds and mattresses in place of horsehair. The seed itself is similar to cotton-seed, in that oil can be expressed from it, such oil being used as a food, and for soap-making in Holland. As will be seen from the analytical constants obtained on the examination of the oil, it is of the same class as cotton-seed-oil. The sample examined by the author possessed the following characteristics:

The oil was of greenish-yellow colour, and slight inoffensive smell and taste. It was viscous and turbid, becoming clear, however, on standing from the separation of stearin, analogous to cotton-seed stearin. When treated with sulphur chloride or sulphuric acid, Kapok-oil evolves considerably more heat than does cotton-seed-oil.

The rise of temperature with sulphuric acid (determined in the manner recommended by Archbutt) is 95° C. Strong nitric acid gives a coloration similar to that produced with cotton-seed-oil, but the reaction takes place more slowly, and the shade is rather green-brown than red-brown. Complete saponification is accomplished with some difficulty. The fatty acids are easily soluble in petroleum ether, and are therefore free from hydroxy acids. The following figures for the usual criteria of an oil are given :

Specific gravity at 18° C.	0.9199
" " of fatty acids	0.9162
Fusing-point " " " "	29°
Solidifying-point of fatty acids	23-24°
Saponification number of oil	181
" " fatty acids	191
Mean molecular weight of " "	293
Iodine number of oil	116
" " fatty acids	108
Hehner's number	94.9

Kapok-oil is, therefore, somewhat more drying in character than cotton-seed-oil.

B. B.

Composition of Pepper. E. v. Raumer. (*Zeit. angew. Chem.*, 1893, 453-457.)

—The adulteration of black pepper with the cortex which has been removed from another portion for making white pepper cannot be detected by the microscope. The author has therefore turned his attention to the composition of the cortex as compared with that of the pure pepper.

For the estimation of starch, 5 grammes of pepper are boiled with 200 c.c. of water for half an hour under an inverted condenser. After the contents of the flask have cooled to 65°, some pure diastase (prepared after Lintner) is added, and the liquid kept at 65° for four to five hours. 25 c.c. of lead acetate are now added, and the whole made up to 250 c.c. After an hour, during which time the flask is intermittently shaken, 200 c.c. are filtered, and a concentrated solution of potassium hydrogen carbonate is added to the filtrate, whereby the excess of lead is precipitated. The liquid is again made up to 250 c.c., 200 c.c. are filtered and neutralized with acetic acid; 20 c.c. of 25 per cent. hydrochloric acid are added, and the liquid finally heated under an inverted condenser for two hours and a half. The starch is now completely inverted, and the reducing value of the solution may be taken.

Cellulose is determined after Henneberg and Stohmann's method.

The moisture in the pepper cannot be determined at 100°, owing to the volatilization of essential oil; even over strong sulphuric acid some of this oil is lost, as evidenced by the darkening of the acid.

The quantity and composition of the ash of black pepper is known to differ considerably from that of white pepper. Black pepper has more ash than white, and this ash contains more alkali. But so wide are the limits of variation that this knowledge is of little value in detecting cortex in black pepper.

The percentage of phosphoric acid in the cortex ash varies from 3.9 to 4.4, while that in the black pepper ash varies from 5.7 to 10.2. Even here, however, admixture leaves the analyst in doubt. As a rule, the specifically lighter sorts of pepper

approach cortex so nearly in content of phosphoric acid and alkali that the better sorts of pepper can be mixed with cortex and be disposed of as lighter sorts.

The same remarks have, unfortunately, to be made of the content of cellulose and starch. Calculated on the ash-free substance, the highest percentage of cellulose in pure pepper is 25.1; the lowest in cortex is 29.1. The highest percentage of starch in cortex is 16.5; the lowest in pepper is 24.0. In pure black pepper the cellulose will vary between 11 and 25 per cent.; starch, between 24 and 47 per cent., still calculated on the ash-free substance. The starch falls in percentage as the cellulose rises.

It would seem as though these figures were sufficiently dissimilar to permit of the immediate detection of admixture of cortex to black pepper. Yet this is not so. Witness a sample, known to be a mixture, which gave a high ash and alkali content, but so much as 25.6 per cent. of starch, and so little as 19.7 per cent. of cellulose, calculated on the original substance, or 30 per cent. and 23 per cent. respectively calculated on the ash-free substance. Such figures for starch and cellulose are well within the limits for black pepper given above.

The author hopes that the estimation of the alcohol (?) extract of the sample may help in the detection of cortex fraudulently present, for this portion of the peppercorn contains more oil than the kernel does.

The following figures illustrate the author's points:

	Penang. Per Cent.	Penang. Per Cent.	Singapore, sifted. Per Cent.	Lamong. Per Cent.	Penang, lightest sort in London Market. Per Cent.
Water yielded to H_2SO_4 ...	10.016	10.000	13.306	14.170	9.970
Ash ...	4.107	4.200	3.510	5.035	5.732
Ash insoluble in H_2O ...	2.923	2.380	1.753	3.113	2.995
Ash insoluble in HCl ...	1.075	0.922	0.290	0.740	1.062
Phosphoric acid in ash ...	9.58	9.940	9.990	7.490	7.340
Cellulose in dry, ash-free substance ...	11.47	13.130	18.650	18.320	22.580
Starch in dry, ash-free sub- stance ...	46.90	46.250	32.080	27.410	28.060

	Pure Cortex. Per Cent.		Cortex as separated by Machine. Per Cent.		White Pepper Dust obtained during decortication.* Per Cent.	
Water yielded to H_2SO_4 ...	10.10	6.21	10.56	9.42	11.44	7.77
Ash ...	6.51	7.06	7.05	8.12	9.37	3.82
Ash insoluble in H_2O ...	3.25	3.62	2.41	3.71	5.21	2.51
Ash insoluble in HCl ...	0.56	0.81	0.34	1.33	2.43	0.34
Phosphoric acid in ash ...	3.96	3.69	4.47	4.09	3.70	12.76
Cellulose in dry, ash-free substance ...	34.86	34.52	29.10	34.88	36.81	11.68
Starch in dry, ash-free sub- stance ...	16.57	13.49	15.34	10.28	7.87	43.68

A. G. B.

* This resembles smaller sorts of white pepper both microscopically and macroscopically. It contains cells rich in starch granules.

The Products of the Sublimation of Arsenic. J. W. Rettgers. (*Zeit. anorg. Chem.*, 1893, iv. 403; through *Chem. Zeit.*)—The significance of arsenical mirrors both in toxicology and in ordinary blow-pipe testing has induced the author to investigate the modifications of arsenic that may occur in this form. He has arrived at the following conclusions:

There is no amorphous form of arsenic, the variety generally thus called being crystalline. Two modifications can be distinguished; they are a hexagonal silver-white variety possessed of metallic lustre, and being both specifically heavier and also less volatile than the second kind, which is black in colour, crystallizes apparently in the regular system, and constitutes the true arsenic mirror. The former modification corresponds to red hexagonal phosphorus (red phosphorus having been recently proved by the author to be crystalline), and the latter to yellow phosphorus, which crystallizes in the regular system. Both modifications of arsenic are perfectly opaque; deposits which are yellow or brown, and more or less transparent, consist of the suboxide and hydride, As_2O and AsH . The brown spot on porcelain produced by contact with a flame of arseniuretted hydrogen is not a thin film of As, but one of the brown solid hydride AsH , formed by the decomposition of AsH_3 . This view is confirmed by the fact that arsenic sublimed in an indifferent gas (e.g., CO_2) is deposited in one or other of the modifications described above, the brown transparent product being obtained only in the presence of H or O. Moreover, pure arsenic is insoluble in all solvents, whereas the film on porcelain (AsH) is soluble in many solvents, including hydrocarbons of the benzene series (e.g., xylene), warm methylene iodide and hot caustic potash.

B. B.

Furfural as a Reagent for Sesame-Oil in other Oils. V. Villavecchia and G. Fabris. (*Zeit. angew. Chem.*, 1893, 505, 506.)—It has been pointed out by other observers that the colour reactions obtained by treating oils, albuminous substances, alkaloids, etc., with sugar and sulphuric acid are really reactions of furfural with these compounds.

By saponifying sesame-oil with baryta, and extracting the soap with alcohol and petroleum ether, the authors have succeeded in isolating from sesame-oil: (1) a higher alcohol, $\text{C}_{25}\text{H}_{44}\text{O}$, melting at 137° , and having a specific rotation of $[\alpha]_D^{20} = -34^\circ 23'$ ($C = 5.013$); (2) a crystalline substance, which they call *sesamin*, and which melts at 123° and has a specific rotation of $[\alpha]_D^{20} = +68.36$ ($C = 24.45$); (3) a viscid, non-crystallizable, nitrogen-free oil, which contains the substance to which the colour reactions of sesame-oil are due.

The substitution of furfural for sugar and sulphuric acid in testing for sesame-oil is effected as follows: 0.1 c.c. of a solution of 2 grammes of furfural in 100 c.c. of alcohol is placed in a test-tube, and 10 c.c. of the oil to be tested are added, followed by 10 c.c. of hydrochloric acid (sp. gr. 1.19). The tube is shaken for half a minute, and allowed to stand. Should the oil contain sesame-oil, even less than 1 per cent., the aqueous liquid which settles in the tube will be coloured carmine-red. Instead of 10 c.c. of acid, 1 c.c. may be employed, and the mixture shaken with 10 c.c. of chloroform until the oil has all dissolved; the red colour will appear in the aqueous layer. In the absence of sesame, olive-oil gives no colour save a dirty-yellow tint; a rancid olive-oil may give a greenish colour.

Experiment with a large number of seed, fish, and mineral oils convinces the authors of the certainty of the test.

A. G. B.

**BAKING POWDER NOT AN ARTICLE OF FOOD.
HIGH COURT OF JUSTICE.—QUEEN'S BENCH DIVISION.**

Before MR. JUSTICE HAWKINS and MR. JUSTICE LAWRENCE.

(*Reprinted from the "Times."*)

JAMES V. JONES.

THIS was an appeal from the Quarter Sessions of Glamorgan, affirming a conviction of the appellant by four justices on February 15, 1893, for that he on December 10, 1892, unlawfully did sell to Jones, an inspector under the Foods and Drugs Act, a certain article of food—to wit, baking powder—which had been mixed with a certain ingredient—to wit, alum—injurious to health. The appeal was heard on December 18 last, and

Mr. Justice Hawkins, in delivering the considered judgment of the court, said : The facts seem to be that on December 10, 1892, James sold Jones a packet of Excelsior Baking Powder, for making light and wholesome pastry and puddings, etc., without yeast. On the wrapper were directions as to how much to use, and how to mix it. The object of using the powder was to diffuse carbonic acid gas through the dough, so as to cause it to expand and render the bread, cake, or pastry when baked light and digestible. The baking powder contained 20 per cent. bicarbonate of soda, 40 per cent. alum, and 40 per cent. ground rice. Carbonic acid gas is contained in the bicarbonate of soda, and to liberate this gas properly a chemical combination of an acid with the bicarbonate of soda is necessary. To effect this combination alum is used, the ground rice being added to keep the two in good condition. The ground rice does not pass off with the gas, but remains in the bread. The alum and the bicarbonate of soda, however, assume another form, in which, when eaten with the bread, they are injurious to health. The Sale of Food and Drugs Act, 1875, 38 and 39 Vic., c. 63, in its preamble recites the necessity for food being sold in a pure and genuine condition. Section 2 enacts that food shall include every article used for food or drink. Section 3 enacts that no person shall mix any article of food with any ingredient so as to render the article injurious to health with intent that the same shall be sold in that state. We are asked to say (1) whether the baking powder is an article of food within the meaning of the statute, and (2) whether James sold it to Jones mixed with an ingredient—to wit, alum—injurious to health. Section 3 creates two offences, mixing and selling, and it is the latter James is charged with. The sale of alum is in itself not an offence, even though the seller may know the buyer is going to mix it with bread, and therefore the fact that the alum is sold mixed with other things does not alter the position. No one would dream of using the ground rice contained in the baking powder as an article of food, as it is part of the mixture, injurious or otherwise. One might as well say that poison and flour in equal parts was "food," because flour is a "food." The test is whether the article when sold is an article of food or not. Though the purchaser, if he afterwards mixes it with intent to sell, may commit an offence, the vendor would not be committing one. We are clearly of opinion that the baking powder is not an article of food, and neither the sale of it, nor the admixture of it with an article of food, unless such article is intended for sale, is an offence. For his own use anybody may use it, and, that being so, it would be strange for the vendor to be liable to penalties. We do not, however, mean to convey it as our opinion that nothing can be deemed to be an article of food unless made up into an eatable or drinkable form, such as flour, butter, salt, mustard, pepper, etc., for although no one would dream of eating them alone, yet they are intended to be consumed by mankind. We think the Court of Quarter Sessions was wrong in treating baking powder as an article of food. It is unnecessary to answer the second question. The order of the Quarter Sessions and the original conviction must be quashed.

Sir R. Webster, Q.C., Mr. Brymnor Jones, Q.C., and Mr. Macmorran were for the appellant; Mr. Finlay, Q.C., and Mr. Rhys Williams for the respondent.

ERRATUM.—In third column of table on page 21 of preceding number, for "Oxygen absorbed in four hours" "0.45" read "0.045."

THE ANALYST.

MARCH, 1894.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on February 7th at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner occupied the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed as members: Leonard Archbutt, F.I.C., 11, Charnwood Street, Derby; B. E. R. Newlands, 27, Mincing Lane, E.C.; T. H. Pearmain, Apothecaries' Hall, Blackfriars, E.C. As associate: Raymond Ross, Assistant to Dr. Horace Swete.

The following gentlemen were duly elected members: Frederick Dunn, James Edmunds, M.D., A. R. Ling.

Papers were then read on "The Detection of Cotton-seed Oil in Lard," by Edward J. Bevan; "Milk and Milk Products," by H. Droop Richmond; and on a "Suggested Addition to Richmond's Milk Scale," by Walter J. Sykes. The publication of these is unavoidably held over.

PRESIDENT'S ANNUAL ADDRESS.

(Delivered at the Annual Meeting, January 24th, 1894.)

GENTLEMEN,—My first and most pleasing duty is to thank you most heartily for the great compliment which you have paid me by re-electing me as your President for a second year. Some of you are aware that I was anxious to have a successor appointed at the expiration of my first year of office, because I feel that I have not been able to discharge the duties of a President even in a perfunctory manner. Living as I do at so great a distance from our headquarters, and having but little natural capacity to encounter the "horrors of the Channel passage," are my excuses for being an absentee President. My remorse would, however, have been greater than it is had I not been cheered by the reflection that my good friend the immediate past President, Mr. Otto Hehner, had kindly promised to be always present at your meetings and to preside in my absence, if you were willing that he should, which I venture to think was always the case.

Everyone must naturally feel some pride in being placed *primus inter pares*. I assure you that I feel very deeply the honour of being for the time the official head of a profession with which I have been intimately associated almost since the days of my boyhood. During my long and busy life, I have devoted no inconsiderable

proportion of my time to professorial and medical work, yet I have always regarded myself as being primarily and chiefly an analytical chemist. I have indeed lived through the period in which chemical analysis has been formed into a distinct profession. When I was a student, commercial analytical work was almost completely carried on in the laboratories of professors and lecturers. Except in London and a very few other large towns, there were no chemists who exclusively devoted themselves to analysis for technical and commercial purposes. The importation of guano and the establishment of artificial-manure works—events occurring towards the middle of the century—gave a great impetus to the development of chemical analysis into a distinct profession. For a time the examination of fertilizers was conducted in laboratories not connected with works, but very soon the larger artificial-manure manufactories began to secure the exclusive services of analysts. Then it became the practice to subject much more largely than formerly all sorts of things to analysis for commercial purposes—nitre, nitrate of soda, oil-cakes, ores, soils, kelp, drugs, etc., were appraised chemically. The next epoch was the passing of the Act relating to adulteration of food and drugs in 1860. This measure, however, created but little demand for analytical work, and very few public analysts were appointed under its provisions. Dr. Hill, of Birmingham, and myself are the only survivors of the few analysts which the Act of 1860 called into existence. In 1872 a more effective Adulteration Act was passed, and it led to the immediate appointment of a large number of analysts. The sudden demand for them could not be properly met, and no doubt at first many persons were appointed as official analysts who had not the proper qualifications for such a position. It is probable that the somewhat low estimation in which the public analysts were held by the professors of chemistry and the chemists engaged in original research was due to the fact that many of the former were well known to be persons who had no real claim to the designation of either a theoretical or practical chemist. The extremely small salaries which the majority of them received were not likely to elevate them in the estimation of the university and college professors.

Shortly after the passing of the first Adulteration Act greatly increased attention was given to the subject of public health. Year by year sanitary statutes were enacted, and in process of time thousands of medical officers of health were appointed. The analysis of water used for potable purposes now began to be carried out upon a gigantic scale, and although some of the work was performed by the medical officers themselves, by far the larger proportion of it devolved upon the analysts.

It seems pretty clear, then, that it is chiefly to the use of fertilizers and feeding-stuffs, and to the passing of the Acts relating to adulteration and public health, that we are indebted for the creation of the distinct profession of the analytical chemist in these countries. They still furnish the greater portion of material for our work, but steadily the demand upon the skill of the analyst is extending from every direction. There will soon be hardly a commercial article that will not be bought or sold on the certificate of the analyst.

Having thus briefly surveyed the progress which the profession of chemical analyst has made during the last half century, let us now examine into our present position and our prospects.

The analysts of the present day are in status far in advance of their predecessors of twenty years ago. A large proportion of the public analysts originally appointed have disappeared, and their place is now occupied by much more competent persons. The emoluments of the analyst have increased, chiefly because the amount of work has been enormously augmented. The teaching of analytical chemistry, based upon the general principles of chemical science, is now carried on in most of our large towns, and the laboratories of the leading analysts are excellent practical schools for the younger chemists. Science is so vast that we have to subdivide it, so that our finite understandings may be able to grasp even an almost infinitesimal portion of it. Chemistry, itself a fragment of science, cannot by any one mind be studied as a whole; and surely that department of it which relates to the analysis of bodies must be sufficient to afford sole occupation for even the most intelligent mind. The processes of analysis are so numerous, the number of distinct articles to be examined so large, that there seems to be even a necessity for chemists to devote themselves altogether to special departments of analysis, as indeed many of them do at present.

It is pleasant to be able to affirm, without fear of contradiction, that the professional status of the chemical analyst is now fully equal to that of the great majority of the other professions. The training required in order to qualify him for his calling involves an amount of intellectual exertion quite equal to that necessary to attain the position of a barrister or an engineer. The proportion of our professional brethren who proceed to university degrees in arts and science is steadily increasing, and the day I believe is not distant when no one can be appointed to any official position as an analyst who has not passed a qualifying examination for such a situation. Both branches of the legal profession are regulated by law, and no one can practise either without having gone through a prescribed course of instruction and passed examinations. Medical men, dentists, pharmaceutical chemists, and even druggists, who merely sell drugs, have all to pass examinations qualifying them to practise. The plumbers are demanding an Act of Parliament to establish the principle of examining and registering the members of that handicraft. It is clear, then, that the time has arrived for seriously considering the question of the registration of chemical analysts.

It may be said that the Institute of Chemistry affords the machinery for testing the knowledge of chemical students, and for giving those found to be worthy of it the title of Fellow or Associate. Those titles have not, however, as yet been recognised in the same way as those conferred by the legal and medical corporations and other bodies, such as the Pharmaceutical Society. They confer no exclusive privileges. A person who has no chemical title or degree, or diploma in science, may be legally appointed an official analyst. My predecessor, Mr. Hehner, has insisted upon the importance of testing the competency of candidates for the office of public analyst by obliging them to obtain diplomas, and he suggests that our Society would be the proper body to grant such a diploma. I was at first averse to this proposal, because I feared that the Institute of Chemistry might regard it as an interference with their functions as an examining body; but I confess that I have now come to the conclusion that the proposal has much to recommend it, and that it ought at least to be seriously considered. If our Society ever comes to the determination to establish the diploma

of analytical chemist, I trust that the examinations for it may be of the most comprehensive and stringent nature, and that it should not be conferred upon anyone, no matter how eminent as an analyst, who was unwilling to submit to the test of examination. Supposing at some future time that a chemical diploma became a *sine qua non* for a chemical appointment, it might be said that it would not be fair to disqualify an analyst who had no diploma, but whose record proved his competency. This difficulty might be got over by providing that persons who had held official positions as analysts up to a certain date should be eligible. One of the provisions of the last Medical Act declares that no person shall be appointed medical officer of health for any town containing more than 50,000 inhabitants who does not possess a degree or diploma in public health or State medicine, except he has already acted as a medical officer of health for a large town.

The question as to the minimal amount of certain constituents of articles of food, drink, and drugs which should be permitted to pass unchallenged, is one which imperatively requires to be answered in no uncertain voice. The "battle of the standards" is being incessantly waged, not only between the analysts and the vendors of food and drink, but between analysts and analysts. The Legislature has defined the strength of the stronger alcoholic liquors, below which the latter must be regarded as adulterated, unless the strength be declared before purchase. The Legislature should fix similar standards for other articles, particularly beer, milk, buttermilk, cream, butter, cheese, and vinegar. The amount of rectified and proof spirit in tinctures and other drugs might be defined, and perhaps also the amounts of solid matter held in solution. In prosecutions in cases of deficiency of alcohol in tinctures the excuse is invariably set up that the deficiency is due to evaporation during the process of manufacture or during storage.

The maximal amount of water which should be permitted in butter is one which the Legislature should determine as speedily as possible. The monstrous proposition has been made that butter might legitimately contain 10 per cent. of salt and 25 per cent. of water! There are many persons of opinion that no person should be punished under the provisions of the Food and Drugs Act who did not knowingly adulterate the articles they prepared or sold. There is, however, such a thing as criminal negligence, which people have often to pay very smartly for. If a careless driver knocks down and kills or maims a person, it is no excuse that he did not intend to do so. It is quite clear that at all times of the year heavily salted and lightly salted butters are made containing from 8 to 14 per cent. of water. There is no good reason why some persons should make butter containing 20 per cent. or more of water, whilst in the same district, and under similar conditions of climate, other persons make butter containing 10 or 12 per cent. of water. The butter-maker is morally bound to take care that he produces an article of the "nature, substance, and quality" which the public expect, and ought to receive; and if he does not, he certainly deserves the very mild punishment which is permissible under the Food and Drugs Act.

Should a Bill to amend the Sale of Food and Drugs Act be introduced into Parliament, it will be necessary to include in it many amendments of the provisions

of the existing Acts. It seems to me that there is too much red-tapeism in the forms prescribed by the Acts. Many prosecutions have fallen through, for example, because the food inspectors merely informed the vendors that they intended to submit the article purchased to analysis, but did not say by the public analyst, or did not name that functionary. Surely it seems sufficient to intimate to the vendor that the inspector purchased the article with the view of ascertaining whether it was pure or adulterated.

I have known many cases to be dismissed because the articles sent for analysis by parcel post were not registered, though the Court of Queen's Bench in Ireland have unanimously declared that articles for analysis may be sent by railway—a less secure mode than by parcel post.

Over and over again magistrates have dismissed cases because no evidence was submitted showing that the analyst's signature to the certificate was genuine. The Chief Secretary to the Lord-Lieutenant for Ireland has requested the Irish public analysts to put an impressed stamp upon their certificates, in order to give them a more official and genuine appearance.

Private persons who believe that the articles which they have purchased are adulterated, find it difficult to prosecute the vendors. In all such prosecutions the attendance of the analyst appears to be requisite. Until lately prosecutions for the sale of unsound food were practically limited to cases where the articles had been seized by an inspector of nuisances or medical officer of health. The last amendment of the Public Health Act provides that a private person who has purchased an article of food which proves to be unfit for use can hand it over to the sanitary officer, and he can proceed in reference to it in the same manner as if he had himself seized it. Similarly, it might be provided that a person who had reason to believe he had bought an adulterated article could hand it over to be dealt with by the food inspector. In such case, then, the evidence of the analyst, which at present must be parole, might be given in the usual form of certificate. The inspector should, of course, hand a portion of the article to the vendor, and retain a third part for analysis, if so ordered, by the Somerset House analysts. I have had often to go to distant parts of Ireland to give evidence in adulteration cases, in which inspectors were not the prosecutors, but in which there was no sufficient reason, if the law permitted it, to prevent them from acting as such.

I have now to say a few words in reference to the work of our Society, and our losses and gains in 1893.

In a Society composed of two hundred members, many of them, like myself, "in the sere and yellow leaf," we could not expect to pass through the year without some of us paying the great debt we owe to nature. Our losses by death have, however, been comparatively small. There are few communities in which a death-rate of fifteen per thousand living would not be regarded as a small one. Ours has not exceeded that rate. But though there have been only three gaps made in our ranks in 1893, they have been in the strongest parts of our line. We have to deplore the death of Mr. C. W. Heaton, for many years our valued treasurer, and a hard-working member of our Society. His loss will be difficult to make up for. We have

also taken from us, by one of those inscrutable decrees of Providence, in the flower of his manhood, our valued friend, Mr. R. H. Davies, for three years one of our honorary secretaries. Our third loss has been in the person of Dr. H. Critchett Bartlett, who in former years was one of our most energetic members, but who for some time past had practically given up chemical work.

Owing to resignation and to unduly prolonged absence of mind in reference to annual subscriptions, 3 members and 4 associates are no longer summoned by our courteous secretaries to attend our meetings. On the other hand, we have added to our numbers 1 honorary and 17 ordinary members and 11 associates. Our Society now consists of 9 honorary members, 188 members, and 25 associates, making the respectable total of 222.

Dr. Tilden, F.R.S., President of the Institute of Chemistry, is the gentleman elected as honorary member. He deserves the highest compliment that our Society could pay him, for in him the claims of the analytical chemist have perhaps their most eloquent and earnest advocate.

The various papers read before our Society in 1893 are further evidence, were such required, of the activity of our members and their capacity for original research. Many of the papers contain matter of great interest, and on looking through them I cannot discover one which could be fairly regarded as dealing with trivial matters.

The following is a list of the papers :

Jan. 4.—"The Estimation of Insoluble Fatty Acids." By Charles E. Cassal.

Feb. 1.—"The Stock Nitrogen Process: (i.) A Reply to Mr. W. P. Skertchley. (ii.) A Modified Distillation Apparatus." By W. F. Keating Stock.

"On Milk and Milk Products." By H. Droop Richmond.

March 1.—"A Source of Error in the Volumetric Estimation of Chlorides by Silver Nitrate Solution." By W. Gathorne Young.

April 5.—"The Lister Babcock Milk Tester, with some Suggestions for Extending its Use." By G. Embrey.

Mr. J. Baynes showed some analytical appliances of much interest.

May 3.—"Notes from the Aylesbury Dairy Company's Laboratory: (i.) The Action of Heat on Milk. (ii.) Points in the Analysis of Condensed Milk. (iii.) The Adulteration of Milk by Diluted Condensed Milk, and its Detection. (iv.) Points in the Determination of Nitrogen by the Kjeldahl and Stock Processes. (v.) The Boiling Point of Salt Solutions." By H. Droop Richmond and L. K. Boseley.

June 7.—"Sulphuric Acid Hydrolysis of Butter Fat." By Dr. Rideal.

"Notes on Rice Oil and Maize Oil" and "On Rapid Saponification for Analytical Purposes." By Alfred Smetham.

"On Ginger—with Special Reference to Discrimination between Genuine and 'Exhausted' Specimens." By Dr. Bernard Dyer and J. F. H. Gilbard.

"Notes on Centrifugal Milk Testers." By Dr. Vieth.

July 18.—"The Results of the Working of the Sale of Food and Drugs Act in Dublin." By Sir C. A. Cameron, M.D.

- July 18.—“Some Recent Improvements in the Babcock Machine.” By Prof. Tichborne.
- “On Vinegar: its Nature and Manufacture.” By A. H. Allen.
- “Notes on Butter.” By Otto Hehner.
- “On the Detection of Heated Cotton Seed Oil in Lard.” By W. G. Crook.
- “On the Estimation of Chlorine in Certain Waters.” By T. Fairley.
- “Some Simple Appliances for Measuring the Consistency of Semi-Solids; with Note on a New Method of Butter Examination.” By C. E. Sohn.
- Oct. 4.—“On the Examination of Copper Sulphate.” By John Ruffle.
- “Distinction between Iron Pyrites and Oxide of Iron in the Commercial Analysis of Mineral Phosphates.” By H. H. B. Shepherd.
- “On the Discrimination between Genuine Abnormal Milks and Adulterated Milk.” By Mr. H. Droop Richmond.
- Nov. 1.—“Note on an Abnormal Melting Point.” By E. J. Bevan.
- A Discussion took place on the Vinegar Question. Opened by A. H. Allen.
- “On the Estimation of Beef-stearine in Lard.” By W. F. K. Stock.
- Dec. 6.—“An Improvement in Richmond’s Milk Scale.” By Charles E. Cassal and B. Henry Gerrans.
- “Leffmann-Beam Method of Fat Estimation in Milk.” Part III. By H. Droop Richmond and L. K. Boseley.
- “On Vinegar.” By Edward Collens.

Before concluding my brief address I avail myself of the opportunity to thank my friend Dr. Dyer, one of our honorary secretaries, for his kindness and courtesy in keeping me *au courant* with the proceedings and aims of our Society. Living as I do so far from our meeting-place, I fear that Dr. Dyer’s valuable time was often unduly encroached upon by his keeping up so frequent a correspondence with me. His new colleague, Mr. Bevan, will, I am sure, take no umbrage when I say that, although we have the advantage of two secretaries, the most arduous part of the secretarial work has hitherto devolved upon Dr. Dyer. I am glad, however, to find that he has so energetic a colleague in our new secretary, Mr. Bevan.

Gentlemen, our Society has now become firmly established amongst the scientific institutions of these countries. Its members are to be found not only in these islands, but in most parts of our vast colonial empire. We might, indeed, fairly claim to entitle ourselves the British Imperial Society of Analysts. We have done good work in the past, but we have much to accomplish in the future. We share with the medical officers of health the guardianship of the health of the people. As we are the natural enemies of fraud, I trust that our services may be always enlisted on the side of those who strive to establish high standards of purity and quality in the food, drink, and drugs used by the people. For these services the public must surely sooner or later feel grateful, and will not fail to recognise the importance of the rôle of the analyst—they will not deny him the liberal remuneration and social distinctions which so many of the other professions enjoy.

In conclusion, let me avail myself of the occasion to cordially wish our Society and each of its constituents a most prosperous year in 1894.

Mr. Otto Hehner said that the President's address was full of suggestive matter. He did not propose to enter thoroughly into a discussion of the address, although he felt tempted to do so. But there were two or three things which he might touch upon, and one of them was that the experience of those who had practically seen the rise of analytical chemists in this country was very valuable to the younger members of the Society. The brief sketch which Sir Charles Cameron had drawn showed how, barely a generation ago, analytical chemists were almost unknown, while now there were large numbers scattered over the United Kingdom. The competition for the work was great, but new fields opened daily and new problems required solution. Many difficult questions had been more or less settled, but a very large amount of work yet remained to be done, and there was plenty for young chemists to do. The President had referred to what all analysts felt to be a matter of vital importance—the condition of the Food Act in its present form. That Act was looked upon at one time almost with pride, and foreign countries sent emissaries and delegates to study it in England, the place of its birth. It was imitated, and it had been the mother of Food Acts all over the civilized world; but now, when it was almost on the eve of attaining its majority, it was already beginning to exhibit manifest signs of advanced decrepitude; it was so riddled with loopholes that it could barely hold itself together. When he had the honour to occupy the chair in that Society, he had hoped that the time was not far distant—in fact, he thought it had then arrived—when the Act would be subject to proper revision. He thought that public analysts, anxious as they all were to defend the public interests, should be prepared to bring forward a measure which should be better than the Act which was now tottering to its grave. It certainly was not creditable to the country that the Act should be in its present state, and that from want of legislative progress in this direction England should have fallen so far behind other nations. It was a pleasure to him, and a great privilege, to move that the Society accord a cordial vote of thanks to Sir Charles Cameron for his address, and to move that it be printed in *THE ANALYST*.

Mr. Alfred H. Allen said that everyone present had listened with very great interest to the address which Sir Charles Cameron had put before them. Under his presidency the Society had a most successful summer meeting in Dublin, and on that occasion they had presented to them all the lions of the city; and not only had they had an interesting meeting, but that day was followed up by the hospitality of Sir Charles Cameron under circumstances which those present would not readily forget. He fully sympathised with the remarks made by Mr. Hehner as to the necessity of being prepared for the legislation which might be anticipated during next Session of Parliament. The enemies of public analysts were numerous. They had not only to deal with the lawyers, whose professional business it was, as Mr. Hehner had said, to pick holes in Acts of Parliament and get their clients off, however worthy they might be of condemnation, but they had also to deal with the large class of people actually interested in maintaining adulteration and in defeating the machinery of the Acts. Prosecutions frequently failed in consequence of the defence of warranty being

set up, and it should be remembered that Sir Charles Cameron, Bart., M.P., had brought in a Bill in the last Session of Parliament with the express object of making an invoice equivalent to a warranty in all cases. Even at the present time a decision of the High Court laid it down that an invoice of a special kind was to be regarded as a warranty, and that entirely exonerated the retail dealer in the proceedings. Unfortunately, in quarters where analysts could reasonably have expected assistance in carrying out their duties, they met with nothing but rebuffs. There were instances where, unfortunately, the Somerset House chemists had not adopted a mode of doing their duties in a manner which appealed to public analysts. They had, apparently, chiefly in view the protection of questionable trade practices instead of regarding themselves as guardians of the purity of the food supply of the public. The official standards and limits deliberately adopted by the Society of Public Analysts after due consideration should be recognised by law, subject to an appeal to the Privy Council, or some machinery less difficult to set in motion than an Act of Parliament. The Society were by far the fittest body to form opinions as to what the standards of quality of articles of food should be, and on what data they should be judged. He had very much pleasure in seconding the resolution which had been moved by Mr. Hehner.

The motion was carried with acclamation.

OFFICERS AND COUNCIL OF THE SOCIETY OF PUBLIC ANALYSTS FOR 1894.

President—Sir Chas. Cameron, M.D., F.R.C.S.

Vice-Presidents (who have filled the office of President)—M. A. Adams, F.R.C.S.; A. H. Allen; A. Dupré, Ph.D., F.R.S.; Otto Hehner; Alfred Hill, M.D.; J. Muter, Ph.D., M.A., F.R.S.E. (Who have not filled the office of President)—Thomas Stevenson, M.D., F.R.C.P.; Charles E. Cassal; Walter J. Sykes, M.D.

Treasurer—E. W. Voelcker.

Hon. Secretaries—Edward J. Bevan, Bernard Dyer, D.Sc.

Other Members of Council—James Baynes, Sidney Harvey, W. Morgan, Ph.D., W. Cobden Samuel, W. F. Keating Stock, C. R. A. Wright, D.Sc., F.R.S.

The names of those Members of Council whose term of office has not yet expired, and who consequently do not retire this year, are R. Bodmer, W. Chattaway, John Hughes, E. W. T. Jones, H. Droop Richmond, and Alfred Smetham.

THE ANNUAL DINNER.

Immediately after the Annual Meeting the members of the Society and their friends adjourned to the Criterion, where the annual dinner took place. The chair was occupied by the President, Sir Charles Cameron, supported by Captain Verney Cameron, C.B., R.N., Dr. F. Allan, Dr. Abraham, Mr. Otto Hehner, Dr. Muter, Mr. Alfred H. Allen, Dr. Stevenson, Mr. C. E. Cassal, Dr. Alder Wright, Dr. J. A. Voelcker, Mr. J. A. B. Newlands, Mr. B. Newlands, Mr. E. W. Voelcker (Hon. Treasurer), Dr. Bernard Dyer (Hon. Sec.), Mr. Edward J. Bevan (Hon. Sec.), Mr. Aubrey Rake (Solicitor to the Society), etc.

The following toasts were proposed by the President: "The Queen and all the Royal Family;" "The Army, Navy, and Reserve Forces."

Captain Cameron responded for the Navy, and Captain C. E. Cassal and Surgeon-Major Smith for the Army and Reserve Forces.

Dr. F. Allan proposed the toast of "The Society of Public Analysts." The public analyst, he said, was an indispensable official. Some public authorities, apparently, thought that he was only an ornament, and that he was unnecessary. Others looked upon him as a necessary evil that the Local Government Board insisted upon, who should be paid as little as possible, and about whom it was hoped that he would do as little as possible. He fancied that the public knew very little of what a public analyst was, or what his duties consisted in. He thought that if the public and their representatives as a rule knew what public analysts did, the arduous work, and the skill that was required to carry out the intricate processes, they would more highly appreciate his services than was at present the case. The work of the public analyst was becoming more and more intricate as science advanced. As new methods were discovered for detecting adulteration, the adulterator found out new methods for deceiving; he invented more complicated methods of adulteration. It was even asserted, but by suspicious persons, that the cows found methods of getting water into their milk before they were milked. It was high time that the Society should put themselves more prominently before the public. He was sure very few of the public realised the trouble which was required to make an analysis of even the most simple substances, which were so very freely ordered to be analysed. He believed his selection to propose the toast might have arisen from the fact that he was one of the few medical men present who was a public analyst. In coupling the toast with the name of the Society's distinguished President, he could, without the slightest fear of contradiction, congratulate it on having one very specially fitted by nature for the post. He had had the good fortune to serve under him in a body with which they were both connected, and the way in which he performed his duties was one which gained praise and honour on all sides. He was sure that the Society of Public Analysts must be proud to have such a man at their head.

Sir Charles Cameron, on behalf of the Society of Public Analysts, thanked Dr. Allan for the way in which he had proposed the toast. The Society of Public Analysts was a society composed of men who were all earnest in their work, though they were not always very highly paid. He could only say this, that there was no society with whom he felt more enjoyment in mixing than with his colleagues in the Society of Public Analysts.

Dr. C. R. Alder Wright said he felt it was a great honour to be asked to propose the health of the Vice-Presidents of the Society. When he looked back upon the last twenty years, and saw the progress which the Society had made, the difference between the position in which the public analyst now stood and that in which he stood at the earlier period was exceedingly great. This advance was largely due to the Vice-Presidents, many of whom had, in their time, occupied the presidential chair. There were, of course, two kinds of Vice-Presidents—those who had not yet attained to presidential rank and those who had passed it. There were representa-

tives of both on the list before him. It was his good fortune that evening to couple with his toast the names of Dr. Stevenson and Mr. Hehner. As regarded Dr. Stevenson, he need say nothing to commend him to the sympathy of public analysts. They all knew him by his work. Such men promoted the interests of the Society, gradually raised it in dignity, and advanced it so far in the estimation of the public that it might rank with the highest learned society extant. Of Mr. Hehner he need say even less. He was well known, first as an ordinary member, then as a honorary secretary, as a President, and finally as a past and Vice-President. His work spoke for him. He wished to emphasize what he had already said, that the progress which the Society had made was very largely due to his hard and good work in every sense of the term.

Dr. T. Stevenson, in replying to the toast, said that he was very much obliged for the kind manner in which the toast had been drunk, and to Dr. Wright for the exceedingly kindly manner in which he had spoken of the Vice-Presidents and of himself. Although of late years he had taken but very little active part in the Society, yet he could claim that he was one of its founders. He had been associated with it from the first; and, as he looked around, he noticed with melancholy regret the absence of many of his old colleagues among the officers. It was now more than twenty years ago when he, with others, met to found the Society of Public Analysts, a society which had since advanced very much in the estimation of the public, and in that of chemists generally, and this advance was due chiefly to the activity of younger and more active members of the Society. It was always a very great pleasure to him to see his name among those who held office in it; to read the journal of the Society, and to associate himself, so far as his other duties permitted, with the advances of analytical science. Although, as had been said, his own special line lay outside the Society in a great measure, yet he was always very proud to be considered as ranking among those humble people who, while taking very small fees, did excellent work in the service of the public. This Society had done as good work as any other Society of a like standing, and it had a great future before it.

Mr. Otto Hehner said that he would like to add his mite to the remarks which had fallen from Dr. Stevenson at the regret which he felt at the absence of old faces that evening. Some, alas! had gone never to return, but their memories would ever remain fresh in the minds of those who had known them as dear friends. He was, however, glad to see present his old friend Dr. Muter; he regretted the absence of Dr. Dupré, Mr. Adams, and Dr. Hill, but he was sure they were all present in spirit. He remembered when this was a struggling society; he had seen how it gradually grew from youth to adolescence, from adolescence to manhood, and now its position, he was proud to say, was acknowledged even by those who were once its bitterest opponents. He did not mean to say that the fog was even yet altogether dispelled; on the contrary, it still lay over the country. Few people recognized that public analysts did work of the utmost importance. Those who ought to be most grateful to them for safeguarding their interests, ought to be the first to acknowledge that analysts were their protectors and faithful servants. But they did not. He had only quite recently seen in a country newspaper an appeal to public analysts to turn their attention to ascertaining the distinction between the water of the pump and the water

natural to milk, and to leave off harassing honest traders. He also saw the fog, which sometimes came up the Thames Valley, rise over Waterloo Bridge and creep over a certain building close by, and he saw that building and those within it covered by that fog, which seemed to be impenetrable. Still, "hope springs eternal in the human breast," and that hope which had upheld public analysts through the past twenty years would carry them on still further. This was an epoch—the end of a chapter—in the history of the Sale of Food and Drugs Acts. They had had foisted upon them by Act of Parliament a body of referees, with whom they were anxious to work in unison; but, in spite of all their efforts, they had not succeeded. It might in the past have been in some part their own fault—he did not say it had not been; but it had certainly not been entirely their fault that they could not work in harmony with those referees. Although the public might not be aware of the fact, analysts had done a great deal for them. Nor did the various authorities who were the guardians of the public weal recognize adequately the services of the analysts. He knew that an immense amount of work was set before them, and he hoped that they would not cease their efforts, but persevere in the path they had trodden hitherto.

Mr. Alfred H. Allen said he sympathised with and endorsed everything that had been uttered by Mr. Hehner, and he could only say that, as one of the Vice-Presidents of the Society who had passed the chair, he was sorry to think that some of them had been lost to the Society by death and others were absent that evening through circumstances over which they had no control. When he contrasted the small gatherings of eight or ten years ago with the numerously attended dinner of to-day, it showed that the influence and position of the Society of Public Analysts, and the interest taken in the Society, was materially increasing; and if the members would only do justice to themselves, have confidence in their own powers, and stick together, there was no object which they could not achieve. He looked forward to the day when it should not be simply a society banded together by a subscription of a guinea a year, but that it should be the Chartered Society of Public Analysts, officially recognized; that it should have the power—the legalised right—of laying down limits and standards, and possibly methods of analysis, which should be recognized just in the same way that Parliament had for years past legalized the limits of strengths of spirits. Then the Society would be looked up to as the authority—subject, of course, to sanction by the Privy Council or similar body—which should lay down standards and limits and frame definitions. Then there would not be any possibility of a defence being put forward to the effect that beef-fat was necessary in the manufacture of lard, and that malt vinegar was anything which contained malt. Things such as these were public scandals. If public analysts would only stand by each other they would have in the near future the power of settling these matters on a fair foundation, so as to do no injustice to innocent manufacturers and tradesmen or to interfere with harmless trade practices; but, at the same time, they would prevent a great deal of the fraud which went on at the present day, and which caused hundreds of thousands of pounds of loss annually to the people of this kingdom.

Mr. J. Newlands, in a humorous speech, proposed the health of the other officers.

Mr. E. W. Voelcker, in responding, said he felt that he could only express his intention of doing his best for the interests of the Society in endeavouring to fill the place of the late hon. treasurer, Mr. Heaton.

Mr. E. J. Bevan, in returning thanks, said that he felt great difficulty in occupying the position that he did owing to the fact that the late lamented Mr. Davies had filled the office with such complete satisfaction to all the members. He could only hope, in some small degree, to follow in his footsteps.

Dr. Bernard Dyer, in returning thanks, said that he had a message of greeting to give to the Society from Dr. Vieth, who was continuing his labours in milk chemistry at the new dairy station at Hammelin in the Province of Hanover.

Dr. Sykes returned thanks, and gave some statistics regarding the financial aspect of the Journal of the Society.

Dr. John Muter thought he was very lucky in getting the toast he was about to propose assigned to him, because good wine needs no bush; and, when he stated that it was "The Health of the President," he thought he scarcely need say any more. As regarded the presidential duties, of course Sir Charles Cameron, being so much in Ireland, had not been able to attend many of the meetings, but he came over on every important occasion. It was the regret of his life that he was not able to go to Dublin last year, for he understood, from the accounts he had heard, that the members had had a pleasant time.

The President, in replying to the toast, said that he felt most grateful to Dr. Muter for the way in which he had proposed his health and to the members of the Society and the guests for the way in which they had received it. He did feel very proud indeed to be the President of the Society because, as he had already stated in his address that day, although he performed other functions than those of an analyst, yet he had all his lifetime been an analytical chemist. He had also great pleasure in stating that it gave him much gratification to undertake the duty of President.

It had always been a matter of regret to him that more members did not come over to Ireland to attend the meeting of the Society to which Dr. Muter had referred; but he might mention to those who were not present at the annual meeting held that afternoon that he had been asked by another kindred association—the British Medical Association—to deliver the address on public medicine in Bristol; and he hoped that on that occasion the country meeting of this Society would simultaneously take place with the meeting of the British Medical Association; and that the members of the Society would do him the honour of dining with him in Bristol some time towards the end of July. That would be the way in which he would feel most grateful to the members of the Society—to make the next summer meeting as successful a meeting as the one which he had the pleasure of welcoming in July last.

Dr. J. A. Voelcker proposed the toast of "The Visitors," which was responded to by Messrs. B. J. Newlands, Dr. Phineas Abraham, and Dr. James.

Messrs. Septimus Marsland and Walter Fletcher favoured the company with

songs, and several solos were very tastefully rendered on the violoncello by Herr von Mulder. The musical arrangements were under the direction of Mr. Alfred Smythson.

THE LEFFMANN-BEAM METHOD FOR FAT-ESTIMATION IN MILK. PART III.*

BY H. DROOP RICHMOND AND L. KIDGELL BOSELEY.

(Read at the Meeting December 6, 1893.)

In a previous paper (Part I.) one of us suggested that differences between the results obtained by Leffmann and Beam, by Hehner, and by himself were due to diversity in the amount of acid added, the strength of the acid, and differences in the fusel-oil.

Influence of Fusel-Oil.—Five samples of fusel-oil were obtained from different sources; the smell and colour when mixed with hydrochloric acid varied considerably. A milk was taken which gave 3·66 per cent. fat by the Adams method, and Leffmann and Beam determinations were made in triplicate, using bottles for which various factors had to be used to correct scale readings into actual percentages.

No. of Expt.				101	102	103	104	105
Fusel-Oil	A	B	C	D	E
Fat by Leffmann and Beam	1.	3·70	3·62	3·72	3·60	3·62
"	"	"	2.	3·70	3·65	lost.	3·60	3·60
"	"	"	3.	3·73	3·63	3·73	3·61	3·62
Average	3·71	3·63	3·72	3·60	3·61
Number of revolutions re-	quired to produce complete separation			200	100	250	100	100
separation								

These results show that there is a slight difference in various samples of fusel-oil amounting to 0·1 per cent., and also that results are concordant to 0·03 per cent. It is not necessary to multiply these results, but we have several times found the same difference in other experiments. The fusel-oil we use generally is E, and this has a specific rotation $[\alpha]_D$ of 1·7°, and therefore contains about 30 per cent. of the active amyl alcohol. We thought it possible that by the continued action of hydrochloric acid on the amyl alcohol, that amyl chloride, and possibly di-amyl ether (cf. Rogers, *Journ. Chem. Soc.*, lxiii, 1160) might be formed, and that these might influence the results; we find no evidence of this being the case, as freshly-prepared "fusel mixture" gives results which are identical with a "fusel mixture" prepared for some months. A slight error may be introduced by the use of different fusel-oils, but a few preliminary experiments with each lot of fusel-oil will soon settle the question.

Influence of the Strength of Acid Used.—Embrey (ANALYST, xviii, 118) has pointed out that in a somewhat analogous process it is very important to work with a fixed strength of acid; he does not show that the results are influenced otherwise than by a discoloration of the fat by strong acid.

* Part I., *Analyst*, xvii., 144; Part II., *Analyst*, xviii., 130.

We have taken milks and made estimations with five differing strengths of acid with the following results :

	Adams ...	6.10	3.93	3.53	3.49
106.	98.3% acid	6.07	4.00	3.60	3.52
107.	96.8 "	5.89	—	—	—
108.	96.1 "	5.90	—	—	—
109.	95.3 "	5.92	—	—	—
110.	93.9 "	5.76	3.77	3.37	3.30

The Leffmann-Beam results are here the readings of the scale, and are not multiplied by a factor for correction.

With the strongest acid at the ordinary temperature (about 20° C.), the fat was so dark as to be hardly readable when the whole of the acid was added at once; by cooling the acid and milk down to 5° C., or by adding the acid more gradually, the fat came up in a clear light-coloured layer, and could be easily read; no difference in the results was, however, obtained; we have obtained a large number of results pointing to the same differences by varying the strength of acid. The strengths 98.3 and 93.9 are about the extreme variations for acid sold as "pure"; in the previous parts of this paper these extreme strengths were not used, and consequently the very small differences with varying strengths were not detected. No practical inconvenience is felt from the differences with varying strengths of acid, as a few preliminary experiments with each lot of acid and fusel-oil will give the necessary factor to be applied to the bottles to read correct percentages.

It is advisable to test the strength of the acid before use. For this there are five different methods :

- (1) Estimation of the sulphion (SO_4) by precipitation as BaSO_4 .
- (2) Estimation of the acidity by titration.
- (3) Conversion into ammonium sulphate, and weighing as such.
- (4) Deducing the strength from the density at 15° C. (cf. Pickering, *Journ. Chem. Soc.*, lvii., 331, *et seq.*).
- (5) Deducing the strength from the freezing-point.

If the acid is nearly pure, the density determination is by far the most convenient and exact. One of us has published a table for the calculation of percentages (*J.S.C.I.*, 1890, No. 5), but it has the disadvantage of not being applicable to acids of about 97 per cent. strength; it is necessary to dilute these with 3 to 5 per cent. water (or ice); impurities in the acid may make an appreciable difference.

We do not recommend the titration of the acid, as the error of titration is rather large unless done under special conditions, and standard solutions of alkali may be inaccurate to 1 per cent. of their absolute value, unless carefully corrected for temperature and accurately standardized.

The estimation as sulphion, of course, will return as sulphuric acid all sulphates present as impurities. The determination of the freezing-point requires some skill, but in the hands of experienced observers yields very accurate results; the temperature of freezing of ordinary strengths of acids are so low as to render this method generally troublesome.

The conversion into ammonium sulphate is very convenient, but neutral sulphate of ammonium is never obtained, the residue is always acid to methyl orange; if the

acidity be determined by titration and a correction made, the residue ignited and the non-volatile matter weighed, this method gives results which are approximately correct. By estimating the residue upon ignition of the acid and subtracting twice its quantity from the acid deduced from the density, a fair approximation of the strength can be made.

We are in the habit of using the $(\text{NH}_4)_2\text{SO}_4$ method and the density determination, and the corrected results are not very far apart.

Influence of Time and Speed of Whirling.—Variation in the time of whirling from one half to three minutes produced absolutely no change. Variations in the speed from 700 revolutions per minute to 2,000 produced no change. It is very unlikely, then, that any fat should be held in suspension,* as in that case variations in time and speed of whirling would then show differences.

Influence of Temperature.—Dr. Voelcker suggested to us that temperature would make a difference in the results; it does in this respect, that if the milk and acid are at a high temperature when mixed with the higher strengths of acid, the fat is liable to be dark in colour, and is difficult to read. We have very few specific results as to the influence of temperature, but as we find that these tend to show that there is no appreciable difference, and we find that on hot or cold days the Leffmann-Beam results agree equally well with the Adams determinations, we do not attach much importance to this.

We give a table of results obtained; in many cases a weak acid has been employed as low as 93 per cent. H_2SO_4 , and the factor for the acid and fusel-oil has been in these cases calculated from a few preliminary experiments; in others the bottles have read directly the percentages of fat, a stronger acid having been used.

In correcting for errors in graduation of bottles we always multiply by a factor and never add a constant; if the error is simply due to incorrect graduation, the equation calculated from the results for the relation between Adams (A) and Leffmann-Beam (B) should be $A = xB + O$. Were the fat incompletely thrown up, the equation would then become $A = xB + K$, K being the amount of fat retained. We have taken four of our series, and calculated the equation connecting Adams and Leffmann-Beam, by making every pair of results into a simultaneous equation and combining the solutions; we have also calculated the probable error in the constant K. These are:

1. $A = 1.096B - .04 \pm .04$
2. $A = 0.940B - .02 \pm .03$
3. $A = 0.991B + .01 \pm .03$
4. $A = 1.012B - .03 \pm .03$

In each case it is seen that the constant (K) does not exceed the probable error, and it is therefore practically nothing. We have thus experimental proof that no fat is retained in solution, and that the errors of graduation may be compensated by multiplying by a factor.

In order to throw more light on the rôle played by the fusel-oil in the Leffmann-Beam process, we have found it of assistance to compare the results obtained by a

* We find that we obtain the same result on adding the "fusel mixture" after the acid, as when the "fusel mixture" is added first.

process analogous to the Leffmann-Beam with the fusel-oil omitted—the Babcock process.

This has been lately treated of by Stokes, Shutt, Embrey, Babcock, and Tichborne, and our labour in finding suitable results has been greatly increased by the fact that each of these observers to a notable extent contradicts the conclusions of the others. Out of the mass of information we are enabled to pick out thirteen results by Embrey from which we can calculate a relation between Adams and Babcock. Tichborne gives in a footnote some data from which another relation can also be calculated. These are :

$$\text{Embrey } A = \frac{B}{1.013} + .35 \pm .04$$

$$\text{Tichborne } A = \frac{B}{1.11} + .45$$

These two equations only agree in one point, *i.e.*, in showing that there is fat retained by the acid mixture, a conclusion reached independently by Stokes.

It is a well-known fact that when sugar, sulphuric acid, and fat are mixed, combination takes place, with the formation of a compound of red colour. This red colour is noticed when milk and sulphuric acid are mixed. It is an equally well-known fact that when amyl alcohol is substituted for the fat, a compound of a reddish-violet colour is formed. This we always notice in the Leffmann-Beam process.

We hardly venture to form a theory that the retention of fat in the Babcock process is due to the formation of the red compound, but we think we are quite safe in saying that in the Leffmann-Beam process the fusel-oil itself would combine with the acid and sugar before the fat is attacked, and thus the whole of the fat is available for estimation.

The tendency for the fat to enter into combination will be much less in the Leffmann-Beam process than in the Babcock, seeing that in the former the final mixture contains barely 50 per cent. H_2SO_4 ; while in the latter the strength exceeds 64 per cent. H_2SO_4 , and it is kept at a high temperature by the boiling water. That the formation of a compound containing a part of the fat is probably not the whole explanation is shown by the fact that Embrey and Tichborne obtain results showing relations between the Adams and Babcock which are not in accordance, while neither of these relations expresses the results of Stokes and Shutt; while Tichborne confesses that the Babcock is full of hidden errors. In justification of the error of the Babcock process, Embrey has pleaded that his inaccuracies are the results of the bottles being graduated to show the percentages of butter that can be obtained from the milk, and not the percentages of fat; as this statement is at direct variance with that of the inventor of the method, who says, "The test is designed to show the amount of pure butter-fat in the milk, and not the butter which will be made from it," we have ignored Embrey's statement, and have based the relation we have calculated from his results on the assumption that Babcock's statement is the more reliable.

We have deliberately come to the conclusion that it is to the use of fusel-oil that the Leffmann-Beam method owes its marked superiority. The many objections, which seem so apparent to patentees of other methods that they think it unnecessary

to detail them, we have not only been unable after long investigation to discover, but we find them to be, in mathematical language, negative quantities.

We would like to state, lest any purchaser of Leffmann-Beam machines may be afraid to use them on account of Embrey's statements "that they are dangerous, and that it is desirable to cover them in (presumably with a light tin case), because the cups, which are very liable to come off, would be driven through the operator's body," that we have not heard of any accident from this cause, and that in the many hundreds of experiments we have made, nothing untoward has occurred. We do not, however, think that Mr. Embrey meant this part of his paper to be taken seriously, as the absurdity of putting a light tin case as a protection against a projectile of sufficient force to penetrate the body of a human being is manifest to all.

We wish to emphatically state that the Leffmann-Beam method is quite unsuitable to be placed in the hands of inexperienced persons, though in scientific hands it is capable of great accuracy.

We have endeavoured to study one by one the errors of this method, and to show the means of correcting them. That we have been entirely successful we can hardly hope, but as a long experience has failed to reveal other serious errors, we think that those that have escaped our notice are not important. We hope to find time to study the Babcock method, and to see whether the errors mentioned by Tichborne, and shown by the results of other observers, are inherent or adventitious.

SUMMARY OF CONCLUSIONS.

1. As variations in the sulphuric acid and fusel-oil may slightly influence the result, it is well to obtain large quantities of these at a time, and by a few preliminary experiments fix the factor necessary to convert scale-readings into percentages of fat.

2. It is advisable to use the same strength of acid (94-96 per cent. H_2SO_4 is convenient). This is best estimated by converting a known quantity into ammonium sulphate, drying at 100°C ., and weighing; titrating the acidity with $\frac{N}{10}$ baryta solution, using methyl orange as indicator, and then igniting, and weighing the non-volatile matter (correcting for the barium sulphate formed); or by deducing the strength from the density (hydrometers are rarely of sufficient accuracy, and a pycnometer should be used), and correcting by subtracting twice the weight of the non-volatile matter.

3. If it be necessary to perform the experiment when the atmospheric temperature is high, the milk and acid should be cooled beforehand, or should this not be practicable, the acid should be added in small portions (2 c.c.) at a time, and the bottle shaken between each addition, or a weaker acid may be used.

4. About half to three quarters of a minute's revolution at the rate of from 1,200 to 1,500 revolutions a minute has been found the most convenient in practice. The machine has not been found dangerous at this speed; but, if wished, a longer period of revolution at a slower speed is equally efficacious.

SUGGESTIONS FOR PRACTICAL USE.

This method must not be used for accurate work unless the bottles, using the same acid and fusel-oil as are designed for general use, are standardized against the Adams, or other reliable method. If this be done the results can be relied upon to

0.15 per cent., and are nearly always accurate to 0.1 per cent. It can thus be used for milk control by a skilled operator.

A six-bottle machine is large enough for the most extensive laboratories, as with this 30—50 samples an hour, according to the energy of the operator, can be examined.

The total solids and solids not fat can be calculated therefrom by the milk-scale or Hehner and Richmond's formula, *provided the density of the milk be not taken until after an expiration of twelve hours from the time of milking.* Before this time Recknagel's phenomenon is not complete, and calculation of the total solids may lead to serious errors. It is quite useless to recommend this, or any other rapid method of fat estimation, combined with any tables for calculating total solids, to farmers, as they will only be led into error if they attempt to do any more than estimate the fat. The very doubtful value that a knowledge of the amount of solids-not-fat might have to cheesemakers, is also reduced to nothing.

The proper sphere of this method is the laboratory, and for public analysts for sorting their milks, and for chemists engaged in milk control it leaves nothing to be desired.

TABLE OF RESULTS OBTAINED.

No. of Expt.	Adams.	Leffmann and Beam.	Error.
111. ...	3.80 ...	3.80—3.80 ...	—
112. ...	3.58 ...	3.55 ...	— .03
113. ...	3.71 ...	3.75—3.70 ...	—
114. ...	3.61 ...	3.57 ...	— .06
115. ...	3.70 ...	3.72 ...	+ .02
116. ...	2.79 ...	2.77 ...	— .02
117. ...	3.40 ...	3.40 ...	—
118. ...	2.38 ...	2.30 ...	— .08
119. ...	2.60 ...	2.60 ...	—
120. ...	1.44 ...	1.45 ..	+ .01
121. ...	6.21 ...	6.15 ...	— .06
122. ..	3.67 ...	3.65 ...	— .02
123. ...	3.77 ...	3.65 ...	— .12
124. ...	2.32 ...	2.30 ...	— .02
125. ...	2.93 ...	2.80 ...	— .13
126. ...	3.40 ...	3.35 ...	— .05
127. ...	3.44 ...	3.45 ...	+ .01
128. ...	3.21 ...	3.22 ...	+ .01
129. ...	3.19 ...	3.20 ...	+ .01
130. ...	1.87 ...	1.90 ...	+ .03
131. ...	2.65 ...	2.66 ...	+ .01
132. ...	3.00 ..	3.15 ...	+ .15
133. ...	2.99 ...	3.00 ...	+ .01
134. ...	3.51 ...	3.40 ...	— .11
135. ...	3.64 ...	3.55 ...	— .09
136. ...	2.82 ...	2.72 ...	— .10
137. ...	2.72 ...	2.75 ...	+ .03
138. ...	3.50 ...	3.50 ...	—
139. ...	3.53 ...	3.47 ...	— .06
140. ...	3.31 ...	3.30 ...	— .01
141. ...	3.51 ...	3.45 ...	— .06

No. of Expt.	Adams.	Leffmann and Beam.	Error.
142. ...	3.52 ...	3.50 ...	-.02
143. ...	2.26 ...	2.20 ...	-.06
144. ...	1.60 ...	1.50 ...	-.10
145. ...	3.66 ...	3.61 ...	-.05
146.4038 ...	-.02
147. ...	3.86 ...	3.92 ...	+.06
148. ...	4.00 ...	3.97 ...	-.03
149. ...	3.66 ...	3.66 ...	—
150. ...	6.14 ...	6.16 ...	+.02
151. ...	6.10 ...	6.22 ...	+.12
152. ...	4.48 ...	4.46 ...	-.02
153. ...	3.90 ...	4.00 ...	+.10
154. ...	3.64 ...	3.67 ...	+.03
155. ...	3.59 ...	3.63 ...	+.04

APPENDIX.

TABLE FOR DEDUCING THE STRENGTH OF SULPHURIC ACID FROM THE DENSITY.

(Extracted from the *Journ. Soc. Chem. Ind.*)

Density at 15° C. (Water at 15° C. = 1.)

Density.	Percentage of Acid.	Density.	Percentage of Acid.
1.8384 ...	100.00	1.8385 ...	94.38
1.8404 ...	99.50	1.8380 ...	94.20
1.8418 ...	98.98	1.8375 ...	94.03
1.8430 ...	98.08	1.8370 ...	93.87
1.8432 (maximum) ...	97.50	1.8365 ...	93.71
1.8430 ...	96.93	1.8360 ...	93.56
1.8425 ...	96.39	1.8355 ...	93.41
1.8420 ...	96.02	1.8350 ...	93.26
1.8415 ...	95.67	1.8345 ...	93.12
1.8410 ...	95.40	1.8340 ...	92.98
1.8405 ...	95.16	1.8330 ...	92.71
1.8400 ...	94.96	1.8320 ...	92.46
1.8395 ...	94.77	1.8310 ...	92.22
1.8390 ...	94.57		

This method is most unfitted to place in unscientific hands. Even more so is the Babcock process.

DISCUSSION.

Mr. E. J. Bevan said it appeared to him that the authors had gone somewhat too minutely into detail, seeing that they admitted the possible error to be 0.15 per cent. Nevertheless, the paper was full of very useful hints, and it cleared up several points which he had noticed during the use of the process, and he thought it was a very welcome addition to analytical literature. With regard to the danger of the apparatus, he could plead guilty to putting up a thick iron screen himself. When in Dr. Muter's laboratory that gentleman had recommended him to be on the safe side.

Mr. Burford stated that in using the HCl mixture four months old he had obtained precisely the same results as when using the mixture freshly prepared. He also gave his experience of using the small machine, and in obtaining various readings, and referred to the difficulty he found in weather like the present in consequence of the fat actually congealing in the morning before he could read it. What he had to

do finally, was to put the bottles in a vessel of warm water and keep them in that temperature until he could get a reading. It seemed to him that some definite method for reading was desirable. He would like to make a remark with regard to the mixture of diluted acid, the instructions given with the machine of 1 acid to 2 of water, hot mixture, being very vague. At the moment of mixing 105° C. was registered, but if left it would fall below 60°. If they had a temperature so variable as that, something more definite in the directions was needed.

Mr. Cassal said that the machines ought only to be used by trained chemists. It was absolutely absurd to suggest that dairymaids, ploughmen, and persons of similar attainments were capable of dealing with the difficulties which might arise in the use of analytical machines, and in drawing conclusions from the results obtained with them. To suggest that people of this sort should manipulate with sulphuric acid and amyl alcohol, and attempt to draw deductions from the ceremonies they went through, was ludicrous. He was glad Mr. Richmond and Mr. Boseley had put this matter again into definite shape, as he (Mr. Cassal) had done on a previous occasion, and he hoped they would hear no more in that Society about such machines being suitable for use by the untrained and the ignorant.

Mr. Richmond said he always used the acid-mixture for filling up the bottles cold, and kept it in a bottle fitted with a siphon and pinchcock, containing half a litre. He had not found the least appreciable difference between using cold acid or hot. It so happened that his laboratory was always warm, and he had not been inconvenienced by cold days, but he could quite understand that if it were too cold it would be necessary to warm the whole bottle up again. With regard to the temperature for reading, as far as his experience had gone he had not found any trouble. The first, second, third, fourth, or even fifth readings gave exactly the same results. He thought there was a possible explanation why the readings had decreased; that it was not so much due to contraction of fat in cold weather in the neck of the bottle as to the fact that the fat on cooling adhered to the sides, and could not be properly read. It was always better in his opinion to take too much trouble to find out whether there were errors than to take too little trouble. The time was not wasted, even if there were no tangible results. With regard to the danger of the Leffmann-Beam process, he might say that when the apparatus was going full speed he accidentally put his arm right into it and stopped it, and no material injury resulted. He did not think it was a very dangerous process, but Mr. Revan was no doubt right in thinking it best to be on the safe side.

The Addition of Phenolphthalein to Margarine. F. Hart. (*Chem. Zeit.*, 1893, xvii., 1908-1909.)—Soxhlet has proposed, in order to check the adulteration of butter with margarine, which is still extensively practised, that the addition of a small quantity of phenolphthalein to all margarine shall be made compulsory. In opposition to this proposal it has been stated that margarine thus treated becomes uneatable within eight days of its preparation. Experiments at the dairy station at Prenzlau show that such is not the case. The author has therefore endeavoured to ascertain whether any toxic effect can be ascribed to the small quantity of phenolphthalein which it would be necessary to add.

Portions of margarine weighing 50 grammes each were intimately mixed with 2, 4 and 6 milligrammes of phenolphthalein and exposed to the air and light for six weeks, at the end of which time no perceptible alteration in their quality could be detected, and all were perfectly edible. That the phenolphthalein was still capable of serving as a means for the detection of margarine was proved by treating each sample with caustic soda, when a red colour was unfailingly produced. The author then took doses of phenolphthalein, rising from 1 to 20 centigrammes, without experiencing any ill effects. On raising the dose to 0.3 gramme he suffered from diarrhoea, which became more severe when the quantity exhibited was increased to 0.5 gramme. When taken in quantities such as these, phenolphthalein had an unpleasant, bitter taste. Assuming that the quantity proposed by Soxhlet, namely 2.2 grammes per centner of margarine, were used, a toxic dose of phenolphthalein would be contained in 13.6 pounds of margarine. The author strongly advocates the adoption of the regulation.

B. B.

The Use of the Cryoscopic Method for the Examination of Butter. F. Garelli and L. Carcano. (*Staz. sperim. agrar. ital.*, 1893, xxv., 77, through *Chem. Zeit.*)—The authors, having the fact in view that the glycerides present in butter have a lower mean molecular weight than those existing in margarine, have applied the cryoscopic method, now commonly in use for the determination of the molecular weights of substances that cannot be volatilised without decomposition, to the detection of margarine in butter. Using the Beckman apparatus with benzene as a solvent, they have found the constant for the molecular depression to have the value 53. Adopting this figure, pure butter has a mean molecular weight of 696 to 716, while that of margarine ranges from 780 to 883. The method, like most of those at present in use, is only available for the detection of fairly considerable additions of margarine.

B. B.

NOTE BY ABTRACTOR.—This method, though ingenious, is merely an apotheosis of the ordinary determination of the saponification equivalent.

Determination of Free Acid in Solutions of the Salts of the Heavy Metals. F. Hoffmann. (*Chem. Zeit.*, 1893, xvii., 1318, 1319.)—Free acid can easily be determined in solutions of the salts of alkalies and alkaline earths by titration with an alkali, using a suitable indicator, but this method cannot be adopted for solutions of the salts of the heavy metals, as they have an acid reaction even when containing no free acid. The necessity of determining free acid under these conditions becomes especially apparent when solutions of salts that have been used as electrolytes are examined to ascertain what alteration in their acidity has occurred during the passage of the current. Existing methods are comprised under the following heads :

- (1) Titration with alkali until a precipitate appears.
- (2) Titration with alkali, using congo-red paper as an indicator.
- (3) Evaporation of the solution and extraction of the free acid with absolute alcohol.
- (4) Titration with a standard ammoniacal copper solution.
- (5) Determination of the metal and of the total acid, and calculation of the amount of acid corresponding to the metal; the difference between the acid thus calculated and the total acidity giving the quantity existing free.

The method to be described below was devised by the author for ascertaining the acidity of the electrolyte in a copper refinery, and possesses advantages over those quoted above.

When excess of potassium ferrocyanide is added to a solution of copper sulphate containing free acid, copper ferrocyanide is thrown down equivalent to the amount of neutral copper sulphate, and hydroferrocyanic acid equivalent to the free acid originally present is liberated. The only condition necessary to be observed in order to convert this into a quantitative method is that the dilution of the solution shall be sufficient to prevent the decomposition of the hydroferrocyanic acid. The details of the method are as follows: A known quantity of the solution, containing 2 to 3 grammes of copper sulphate, is treated with excess of 2.5 per cent. solution of potassium ferrocyanide, made up to a litre, shaken, allowed to stand, and 100 to 250 c.c. of the clear liquor (according to its acidity) drawn off and titrated with $\frac{1}{10}$ caustic soda solution, phenolphthalein being used as an indicator. The solution of potassium ferrocyanide should be well protected from light, and in adding it to the copper sulphate solution care should be taken to prevent any portion of the turbidity that forms in it finding its way into the precipitating vessel, as the settling of the copper ferrocyanide is hindered thereby. A similar inconvenience arises from the use of a large excess of potassium ferrocyanide. The presence of sufficient ferrocyanide can be recognised by utilizing the property of ferric sulphocyanide of dissolving in ether, which is effected thus: A drop of *neutral* potassium sulphocyanide is added to the liquid to be tested—which, in the case of commercial electrolytes, always contains enough iron for the purpose of the test—and the whole shaken out with ether, which is no longer coloured red, when all the iron exists as ferrocyanide instead of sulphocyanide. Seeing that in a mixed solution of copper and iron salts the former is precipitated first by potassium ferrocyanide, the absence of iron is a proof of the presence of an excess of the precipitant.

As all the heavy metals are precipitable by potassium ferrocyanide, the method is generally applicable for the determination of free acid in solutions of their salts. It is much simplified when used for the salts of metals, which give white precipitates with potassium ferrocyanide, *e.g.*, lead, silver, and zinc. In this case no filtration or decantation is requisite, for the turbid liquid can be titrated directly. Small quantities of "ous" salts, such as ferrous sulphate, do not greatly affect the results, but the method cannot be used when such salts are present in quantity. B. B.

REVIEWS.

MANURES AND MANURING. By C. M. AIKMAN, M.A., B.Sc., F.R.S.E. (William Blackwood and Sons.)

This is a valuable handbook, exhibiting, throughout, the work of a conscientious and painstaking writer, well up in the contemporary agricultural chemistry of Germany and France as well as that of our own country. We had the pleasure some

time since of noticing an "advance" portion of this treatise, published under the title of "Farmyard Manure." The complete work, which is now before us, is written on the same lines, and does not belie the promise of the preceding fragment. The work is, throughout, lightened for the general reader by the relegation to appendices of statistical and tabular matter, reference to which is facilitated by note marks in the general text. The aim of the author has been to provide a text-book on manures midway, in the scope of its treatment, between Professor Storer's elaborate treatise and the smaller and more popular manuals, and he must be admitted to have carried out his by no means easily effected task with a near approach to satisfaction. Mr. Aikman has had, during a number of years past, an exceptional experience as a lecturer on agricultural chemistry, not merely in the comparatively easy work of the class-room, where regular students imbibe their knowledge systematically, but also in the much more difficult work of expounding its doctrines to "popular" or unscientific audiences in isolated and disjointed discourses. This experience lends probability of fulfilment to the hope expressed by himself that the work now under notice may be of especial value to those engaged in teaching agricultural science. At all events, no agricultural teachers or practitioners of agricultural chemistry should fail to provide themselves with it.

JOHNSTON'S ELEMENTS OF AGRICULTURAL CHEMISTRY. From the edition by Sir CHARLES A. CAMERON, M.D. Revised and in great part re-written by C. M. AIKMAN, M.A., B.Sc., F.R.S.E. (W. Blackwood and Sons.)

Mr. Aikman, whose original work on manures we have noticed in the foregoing paragraph, is indefatigably industrious. Simultaneously with the publication of his own volume there issues from the press this new edition—the seventeenth—of one of the most popular technical treatises known to agricultural science. It is thirteen years since Sir Charles Cameron revised and brought up to date the excellent and once unique work of Professor Johnston, and during these thirteen years the rapid progress of agricultural chemistry has rendered even "Johnston and Cameron," as it is familiarly called, an old-fashioned book, that has been calling out with increasing stress for further revision. This revision has been very fairly accomplished by Mr. Aikman, and as the demand for so time-honoured a text-book is sure to be sustained even in the face of many new ones, agricultural students will be grateful to him for his labours. In these pages it is pleasant to note that while the book remains, and must always remain, associated with the name of its earlier editor, Sir Charles Cameron, the present President of the Society of Public Analysts, the present edition is dedicated by its new editor to one of the present honorary secretaries of that society.

PRINCIPLES AND PRACTICE OF AGRICULTURAL ANALYSIS. By HARVEY W. WILEY, Chemist to the U.S. Department of Agriculture. (The Chemical Publishing Company, Easton, Pa., U.S.)

Professor Wiley's name is so well known in connection with the scientific investigations conducted over a large continent under the superintendence, direct or indirect, of a very active and well-organized Government Department, that anything emanating from his pen cannot fail to arouse interest and to command attention. The work under notice is the first monthly part of what promises to be a very comprehensive manual of agricultural chemistry for the use of analysts, teachers and students. The author hopes to complete the work in twenty-four monthly parts at twenty-five cents each, but this may not prove to be possible. The first part, which begins the subject of soils, contains some excellent plates showing the microscopic structure of various rocks.

THE ANALYST.

APRIL, 1894.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on March 7th at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Alfred H. Allen took the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were duly elected: As Members, Leonard Archbutt, Derby; B. E. R. Newlands, Mincing Lane; T. H. Pearmain, Apothecaries' Hall. As Associate, Raymond Ross, Worcester.

The following papers were read: "Notes from the Laboratory of the Royal Agricultural Society of England: (i.) The Occurrence of a Poisonous Leguminous Seed in Indian Peas; (ii.) Analyses of Waters from Wells in close proximity to Churchyards," by J. Augustus Voelcker, Ph.D., B.Sc. "Beef-Stearin in Lard," by Otto Hehner.

THE COMPOSITION OF MILK AND MILK PRODUCTS.

By H. DROOP RICHMOND.

Read at the Meeting February 7th, 1894.

THIS paper is a continuation of the annual reports of the work done in the laboratory of the Aylesbury Dairy Company. The results obtained during 1893 are given in the present communication. (For previous reports see ANALYST, vii. 53; viii. 33; ix. 56; x. 67; xi. 66; xii. 39; xiii. 46; xiv. 69; xv. 44; xvi. 61; xvii. 62; and xviii. 50.)

The total number of samples analyzed during the year 1893 was 30,504; this is a large increase, the numbers having gradually advanced year by year from 8,817 in 1881, when the company's laboratory was first established.

28,487 samples of milk.

1,121	„	cream.
624	„	separated and skimmed milk.
13	„	buttermilk.
122	„	butter.
91	„	water.
46	„	sundry articles.

Of the milk samples, 14,643 were taken from the railway churns, on their arrival at the company's chief dépôt. The bulk of this is distributed with the least possible delay to the customers, a certain portion being, however, utilized for the production

of cream, etc. To control the men employed in delivering the milk, a further 11,479 samples were taken, before, during and after delivery, and analyzed comparatively. In the following table the average monthly results of these analyses are given. The samples taken on arrival, before and during delivery, were almost exclusively analyzed by estimating the total solids and specific gravity, and calculating the fat. A large portion of those taken at commencement of and after delivery were, however, examined by the Leffmann-Beam method, and the total solids calculated. The agreement is equally good between the different series. No cases in which the milk had been tampered with were found.

AVERAGE COMPOSITION OF MILK DURING 1893.

1893.	On Arrival.				Before Delivery.	At Commencement of Delivery.	During Delivery.	After Delivery.
Month.	Sp. Gr.	Tot. Sol.	Fat.	S. n. F.	Tot. Sol.	Tot. Sol.	Tot. Sol.	Tot. Sol.
January	1·0321	12·68	3·85	8·83	12·50	12·60	12·68	12·71
February	1·0321	12·58	3·76	8·82	12·46	12·56	12·57	12·64
March	1·0320	12·52	3·73	8·79	12·46	12·52	12·52	12·60
April	1·0319	12·53	3·75	8·78	12·45	12·40	12·45	12·55
May	1·0319	12·46	3·70	8·76	12·42	12·37	12·40	12·61
June	1·0316	12·37	3·70	8·67	12·30	12·29	12·31	12·44
July	1·0311	12·41	3·83	8·58	12·31	12·35	12·25	12·39
August	1·0314	12·54	3·89	8·65	12·46	12·47	12·44	12·48
September	1·0316	12·80	4·06	8·74	12·64	12·63	12·62	12·66
October	1·0317	13·20	4·39	8·81	12·93	12·94	13·03	12·99
November	1·0321	13·09	4·21	8·88	12·90	12·91	12·96	12·90
December	1·0321	12·94	4·08	8·86	12·77	12·72	12·76	12·73
Average	1·0318	12·68	3·91	8·77	12·55	12·56	12·58	12·64

After the abnormal summer of 1893, one would have naturally expected to find that the milk this year was of poorer quality than usual; and it will probably be a matter of surprise that the yearly average of total solids is but 0·03 per cent. lower than that of last year, the amount of fat being absolutely identical with that of the past two years, the difference being only in the solids-not-fat. During the months of June, July and August the solids-not-fat were distinctly below the average, and this seems to have been the only effect of the season on the composition of milk. I have already shown (ANALYST, xviii. 270) that by careful examination it has been possible in many cases to distinguish between abnormal and watered samples, and have nothing further to add here on this subject. I may say that when the mixed milk of a whole farm has been examined during 1893, in but one instance has the fat fallen below 3·0 per cent., and in this one case it was 2·99 per cent. The experience of this year has gone to show that the Society's limit for fat is well fixed.

The highest percentages of total solids and fat occurred in October, and the lowest in June.

Cream samples were taken before and during delivery. The average of the results is given in the following table :

AVERAGE AMOUNT OF FAT IN CREAM DURING 1893.				
Month.			Before Delivery.	After Delivery.
January	45.0	45.5
February	45.5	46.0
March	46.3	46.5
April	47.3	48.4
May	48.4	48.9
June	48.8	48.4
July	46.7	47.2
August	47.3	47.5
September	50.8	50.8
October	49.8	50.9
November	46.8	47.4
December	47.3	47.7
Average	47.5	47.9

Considering the difficulty of drawing average samples of cream of such richness, the agreement between the two series is satisfactory.

The average composition of clotted cream (51 samples) was as follows :

				Average.
Water	30.77
Fat	61.49
Ash60
Solids-not-fat	7.74

These figures do not greatly differ from those found in former years.

The bulk of samples of separated milk contained less than .3 per cent. of fat ; in rare instances it contained above .4 per cent. The lowest amount of fat contained in skimmed milk was .04 per cent.

Butter had the following composition :

French butter, fresh ; 35 samples.

Water	15.53 to 11.12 ; average 13.65
Fat	87.06 „ 83.27 „ 84.99
Solids-not-fat	1.86 „ .86 „ 1.36
Salt25 „ .05 „ .13
Reichert-Wollny figure	27.5	„ 32.3	„ 30.2

French butter, salt ; 28 samples.

Water	13.49	9.17	11.61
Fat	88.07	82.74	85.08
Solids-not-fat	4.83	2.32	3.31
Salt	2.95	1.08	1.86
Reichert-Wollny figure	27.5		33.0		29.8

English butter, salt; 43 samples.

Water	15.33 to 10.28	average	12.76
Fat	87.24 „ 82.25	„	84.53
Solids-not-fat	3.94 „ 1.16	„	2.71
Salt	3.31 „ .59	„	1.90
Reichert-Wollny figure	21.6	„	32.3	„	28.1

Swedish butter, salt; 3 samples.

Water	14.36 „ 13.10	„	13.64
Fat	83.52 82.02	„	82.96
Solids-not-fat	3.62 3.19	„	3.40
Salt	2.36 2.07	„	2.19
Reichert-Wollny figure	29.5	31.2	30.5		

One sample of English butter gave a Reichert-Wollny figure of 21.6 c.c.; this was churned at Bayswater, and duplicate determinations were made. This figure is not, however, lower than has been found by Vieth, Mayer, Lupton, and others.

For the discussion of the amount of water in butter, see ANALYST, xx. 16.

The Leffmann-Beam glycerol method has been exclusively used during 1893 for the determination of the volatile acids. It will be noticed this year that French butters give a higher Reichert-Wollny figure than English butters; reference to previous reports will show that this has been invariably the case.

A sample of butter which had been kept for two years exposed to air and light, and which Dr. Vieth had found when fresh to require 29.9 c.c. $\frac{N}{10}$ alkali to neutralize the volatile acids from 5 grammes, took 29.5 c.c. $\frac{N}{10}$ alkali.

Another sample received by Dr. Vieth from the German Commission on Butter Analysis in 1888, and which he has on several occasions analyzed (see ANALYST, xiv. 148; xv. 173), took 33.9 c.c. $\frac{N}{10}$ alkali, as against 32.0 c.c. originally.

A sample of milk was skimmed in three portions—i.e., after six hours' standing, eighteen hours, and twenty-four hours. The last, very small quantity of cream on being churned into butter, took identically the same amount of $\frac{N}{10}$ alkali, 26.5 c.c., as the earlier portions. It seems that the larger and smaller globules of fat do not differ chemically.

The specific gravity of every sample of milk which comes into the laboratory is taken. This determination is of the greatest value, and, when taken with a lactometer, as is our practice, next to no trouble. Lactometers, which can be read to the fourth place of decimals with a little practice, are now made which require only 2 oz. or 4 oz. of milk. It must, however, be borne in mind that the specific gravity of milk cannot be accurately taken until the expiration of some hours after milking, for until this time, as Vieth has pointed out (ANALYST, xiv. 71), Recknagel's phenomenon is still going on.

An opportunity occurred this year of studying this. The following figures were obtained:

Specific gravity	1½ hours after milking	1.0310
„	„ 3¼ „ „ „	1.0322
„	„ 18 „ „ „	1.0325

This rise in specific gravity must not be confounded with a similar rise in the observed specific gravity shown when frothy milk is allowed to stand; it is quite

independent, and is due to some change in the dissolved constituents of the milk. When milked, air-bubbles are always mixed with the milk, but these rise to the top in the course of an hour or less, leaving a very little froth on the top, which is very persistent, and must be removed before taking the specific gravity; occasionally it happens that a sample for analysis contains much froth, and great care must then be exercised in taking the specific gravity.

For very exact determinations of the specific gravity, a small (10 c.c.) Sprengel tube must be used; a bottle is inadmissible, as there is not time for the temperature to become equal throughout before some of the cream separates. With the Sprengel tube uniformity of temperature is reached in a few minutes, and accuracy to '0001 is easily attained.

All exact determinations of total solids have been done by the asbestos method, and the results have been most satisfactory. Macfarlane's chrysotile (ANALYST, xviii. 73) method has also been studied, and the statement in his paper, that this method gives lower results than drying in a basin, confirmed.

It seemed to me that, as no figures were given as to whether the chrysotile could be dried to constant weight, and whether it gave anything to ether, it was desirable to examine into this point:

Weight of tube and chrysotile after 20 hours at 100° C.	...	16.266
" " " " 5 " more	...	16.263

The tube was then wetted with 10 c.c. of water and dried:

Weight of tube and chrysotile after 19 hours at 100° C.	...	16.2675
" " " " 2½ " more	...	16.264
" " " " 17 " "	...	16.269
It was then extracted with ether, and on drying	...	16.263

No ether extract whatever was obtained.

The weighings, though not entirely satisfactory, show that chrysotile can be dried and re-dried to a fairly constant weight.

In three milks the total solids were estimated:

Chrysotile method	...	5 hours at 100° C.	13.00		
" "		20 " "	12.63	12.85	
" "		25 " "	12.63	12.82	12.77
Asbestos method in basin	...	" " "	13.23	13.21	13.09
Difference60	.39	.32

In each case the chrysotile method was lower, and the residue was of a brown colour, while it was white in the asbestos method.

Fat was also estimated by the chrysotile method, and compared with the Adams method:

Chrysotile method—Fat, direct weighing	...	4.12	4.28	3.88
" " " " by difference	...	4.13	4.27	3.91
Adams method	...	4.08	4.17	4.06

The tubes were percolated with ether about six times; in the first two cases a little chrysotile had run through, which makes the results high. There does not seem to be much difference between the two methods.

As the total solid determination does not compare with the methods used in

England, and, moreover, is probably low, as the residue is brown, this method is not likely to be adopted here.

Numerous comparisons between the Adams and Werner-Schmid methods were again made this year; the Werner-Schmid averaged .03 higher than the Adams, and had a probable error of $\pm .041$, the probable error of the Adams being $\pm .027$.

This shows that the Werner-Schmid method is good enough for ordinary work; it has not, however, in my hands proved such a very rapid method, an hour and a half being the least time in which a satisfactory estimation can be performed. I find that it is necessary to allow the tube in which the dissolved milk and ether have been shaken up to stand *at least* ten minutes before drawing off the ether, as before that time the ether contains minute water globules, visible to the naked eye, and more easily seen with a magnifying glass. I have found it very difficult to prevent a slight loss of ether on taking out the stopper of the tube. I have now quite discarded this method for either the Leffmann-Beam centrifugal method, in case a rapid determination is wanted, or the Adams method, where accuracy is a desideratum.

In doing an accurate Adams estimation, attention to the following points is necessary:

- i. The ether must be anhydrous (dried over calcium chloride and distilled is sufficient).
- ii. Schleicher and Schüll's fat free papers should be used, and one should be extracted without any milk on it, in a flask used as a tare for the others.
- iii. Four or five hours' extraction is necessary.
- iv. The coils must be well dried before extraction.

Neglect of the first, second, and fourth precautions causes the results to be too high, and as one or more of these had been neglected in the data from which the formula of Hehner and myself, and my modified formula, were calculated, I thought it desirable to calculate a new formula.

The determinations were made as follows:

Specific gravity. By a Sprengel tube, frequently tared, at 15.55° C. (determined by a Kew standardized thermometer read to $\frac{1}{10}^{\circ}$ C.).

Total solids. Asbestos method.

Fat. Adams method, using the precautions enumerated.

The results are given in two series. In Series I. all determinations of total solids and fat are means of well agreeing duplicates; in Series II. some of the specific gravities were taken with a lactometer, and many of the total solid and fat determinations were not done in duplicate.

Series I. extends over an entire year, and the results are arranged in order of date; the importance of this will be realized later:

SERIES I.

	G.	G. D.	T.	F.	F. calc.	Difference.	Remarks.
1.	34.9	33.7	9.27	.28	.35	+ .07	1st Period
2.	30.8	29.9	12.75	4.10	4.08	- .02	"
3.	33.5	32.4	12.88	3.70	3.65	- .05	"
4.	32.0	31.0	12.83	3.85	3.91	+ .06	"
5.	31.7	30.75	13.09	4.13	4.18	+ .05	"

	G.	G. D.	T.	F.	F. calc.	Difference.	Remarks.
6.	32.2	31.2	12.56	3.65	3.64	— .01	1st Period.
7.	26.0	25.35	10.17	2.92	2.93	+ .01	
8.	32.3	31.3	12.96	3.92	3.96	+ .04	
9.	32.5	31.5	12.75	3.72	3.74	+ .02	
10.	14.1	13.9	5.57	1.56	1.60	+ .04	
11.	31.7	30.75	12.25	3.56	3.48	— .08	"
12.	32.0	31.0	12.48	3.66	3.63	— .03	
13.	31.8	30.8	13.74	4.64	4.72	+ .08	
14.	32.6	31.55	13.04	3.99	3.97	— .02	
15.	25.8	25.15	10.06	2.93	2.88	— .05	
16.	32.3	31.3	12.52	3.60	3.59	— .01	"
17.	32.4	31.4	12.58	3.65	3.62	— .03	
18.	32.0	31.0	13.09	4.06	4.12	+ .06	
19.	29.4	28.55	12.06	3.78	3.82	+ .04	
20.	29.9	29.05	12.64	4.13	4.18	+ .05	
21.	61.4	57.85	20.84	4.73	4.61	— .12	Concentrated Milk.
22.	36.0	34.75	11.68	2.06	2.13	+ .07	
23.	32.8	31.75	12.44	3.48	3.42	— .06	
24.	32.7	31.65	12.42	3.46	3.43	— .03	
25.	32.6	31.55	12.41	3.51	3.44	— .07	
26.	32.7	31.65	11.91	3.01	3.00	— .01	2nd Period.
27.	32.5	31.5	12.32	3.46	3.38	— .08	
28.	23.4	22.85	9.82	3.21	3.18	— .03	
29.	33.2	32.15	11.86	2.92	2.86	— .06	
30.	32.4	31.4	12.59	3.64	3.63	— .01	
31.	32.7	31.65	12.51	3.61	3.50	— .11	"
32.	32.6	31.55	12.58	3.70	3.58	— .12	
33.	32.8	31.75	12.62	3.67	3.58	— .09	
34.	32.6	31.55	12.29	3.41	3.34	— .07	
35.	32.4	31.4	12.25	3.49	3.35	— .14	
36.	32.5	31.5	12.30	3.46	3.37	— .09	"
37.	32.5	31.5	12.30	3.52	3.37	— .15	
38.	31.8	30.8	12.59	3.76	3.76	—	
39.	32.4	31.4	11.91	3.14	3.05	— .07	
40.	32.3	31.3	12.27	3.48	3.38	— .10	
41.	32.0	31.0	12.64	3.80	3.75	— .05	"
42.	30.9	29.95	11.34	2.88	2.90	+ .02	
43.	32.1	31.1	12.60	3.65	3.70	+ .05	
44.	32.0	31.0	12.16	3.35	3.35	—	
45.	32.9	31.85	11.97	2.85	3.01	+ .16	
46.	31.6	30.65	12.51	3.67	3.72	+ .05	3rd Period.
47.	31.6	30.65	12.62	3.77	3.81	+ .04	
48.	32.1	31.1	12.39	3.40	3.52	+ .12	
49.	32.0	31.0	12.42	3.44	3.58	+ .14	
50.	32.1	31.1	12.14	3.21	3.32	+ .11	
51.	32.1	31.1	12.06	3.19	3.25	+ .06	"
52.	32.6	31.55	11.74	3.00	2.88	— .12	
53.	32.4	31.4	11.73	2.99	2.92	— .07	
54.	31.0	30.05	11.95	3.31	3.38	+ .07	
55.	31.5	30.55	12.36	3.51	3.62	+ .11	
56.	31.4	30.45	12.30	3.52	3.59	+ .07	

	G.	G. D.	T.	F.	F. calc.	Difference.	Remarks.
57.	31.3	30.35	12.47	3.76	3.75	- .01	3rd Period.
58.	31.3	30.35	12.53	3.77	3.80	+ .03	"
59.	28.7	27.9	9.72	2.05	2.00	- .05	"
60.	32.0	31.0	12.67	3.75	3.77	- .02	4th Period.
61.	31.9	30.9	12.61	3.73	3.75	+ .02	"
62.	32.0	31.0	12.69	3.77	3.79	+ .02	"
63.	31.8	30.8	12.89	3.93	4.01	+ .08	"
64.	32.2	31.2	12.39	3.53	3.50	- .03	"
65.	32.2	31.2	12.42	3.49	3.52	+ .03	"
66.	33.3	32.2	12.84	3.64	3.66	+ .02	"
67.	33.3	32.2	12.82	3.59	3.64	+ .05	"

SERIES II.

68.	33.4	32.3	12.10	3.05	3.02	- .03	Carter Bell's Method.
69.	31.5	30.55	11.54	2.85	2.93	+ .06	
70.	7.2	7.15	32.50	25.67	25.55	- .12	
71.	32.0	31.0	13.35	4.34	4.32	- .02	
72.	33.0	31.95	13.23	4.08	4.03	- .05	
73.	32.0	31.0	13.21	4.17	4.22	+ .05	
74.	32.6	31.55	12.06	3.16	3.15	- .01	
75.	32.6	31.55	13.07	4.08	3.99	- .09	
76.	32.7	31.65	12.61	3.48	3.58	+ .10	
77.	32.4	31.4	12.92	3.84	3.91	+ .07	
78.	32.4	31.4	13.17	4.15	4.12	- .03	
79.	33.5	32.4	11.83	2.76	2.77	+ .01	
80.	31.6	30.65	15.40	6.19	6.12	- .07	
81.	29.6	28.75	11.30	3.02	3.12	+ .10	
82.	33.5	32.4	11.27	2.22	2.30	+ .08	
83.	29.7	28.85	15.00	6.32	6.19	- .13	
84.	32.3	31.3	12.68	3.76	3.72	- .04	
85.	33.2	32.15	12.03	3.03	3.00	- .03	
86.	33.3	32.2	12.44	3.35	3.32	- .03	
87.	32.3	31.3	11.93	3.11	3.10	- .01	
88.	31.2	30.25	11.30	2.98	2.80	- .18	
89.	33.5	32.4	12.66	3.56	3.47	- .09	
90.	34.2	33.05	12.93	3.68	3.55	- .13	
91.	33.5	32.4	12.20	3.24	3.09	- .15	
92.	33.5	32.4	11.83	2.79	2.78	- .01	
93.	33.1	32.05	11.30	2.38	2.41	+ .03	
94.	32.8	31.75	13.05	3.85	3.93	+ .08	
95.	32.4	31.4	12.26	3.43	3.36	- .07	
96.	31.5	30.55	12.71	4.02	3.91	- .11	
97.	33.9	32.8	10.54	1.44	1.61	+ .17	
98.	33.8	32.7	11.21	2.26	2.19	- .07	
99.	32.9	31.85	12.41	3.39	3.37	- .02	
100.	32.5	31.5	12.49	3.50	3.52	+ .02	
101.	32.2	31.2	11.97	3.18	3.15	- .03	
102.	32.1	31.1	12.13	3.26	3.31	+ .05	
103.	32.5	31.5	12.04	3.06	3.15	+ .09	
104.	28.9	28.1	15.01	6.21	6.36	+ .15	
105.	33.5	32.4	11.27	2.32	2.31	- .01	
106.	30.0	29.1	11.37	2.93	3.11	+ .18	

	G.	G. D.	T.	F.	F. calc.	Difference.	Remarks.
107.	33.0	31.95	11.49	2.65	2.58	-.07	
108.	32.0	31.0	12.24	3.51	3.42	-.09	
109.	30.5	29.6	12.14	3.64	3.65	+.01	
110.	32.5	31.5	11.68	2.82	2.85	+.03	
111.	28.0	27.25	10.63	2.72	2.90	+.18	
112.	29.4	28.55	11.84	3.50	3.62	+.12	
113.	28.6	27.8	11.62	3.53	3.60	+.07	
114.	35.6	34.4	11.64	2.26	2.18	-.08	
115.	33.3	32.2	10.57	1.60	1.77	+.17	
116.	36.0	34.75	9.45	.27	.27	—	

The formula deduced from these determinations was $T = .2625 \frac{G}{D} + 1.2 F$.

It is hardly necessary to state that the maximum specific gravity was used in the calculation.

In this formula it has been assumed that the specific gravity of butter fat at 15.55° C. is .93, and not .94, which was deduced from previous work (*cf.* ANALYST, xiv. 121). A formula was also calculated using .94 as the specific gravity of butter fat, but the agreement with this latter formula was not quite so good. It is evident that the figure .94 is not accurate, though possibly it is correct for the fat extracted by the less accurate methods hitherto used.

The form of formula adopted assumes that the influence of 1 gramme of solids-not-fat dissolved in 100 c.c. is a constant—*i.e.*, that milk may be regarded as a mixture of water, fat and solids-not-fat. It is well known, however, that few substances conform absolutely to this law. To show that in milk this law holds with practical exactitude, I quote the results of an experiment performed some years ago, and published in a different form in THE ANALYST, xiv. 127.

A poor separated milk was diluted, and the total solids and specific gravity estimated in the various mixtures.

Total Solids per cent.	Specific Gravity at 15.55° C.	Influence of 1 gm. of Total Solids per 100 c.c. on Specific Gravity.
9.280	1.03544	3.688
8.758	1.03343	3.693
8.318	1.03170	3.694
7.777	1.02950	3.684
7.456	1.02829	3.690
6.455	1.02439	3.688
Average 3.6895 ± .0033		

From the probable error of the methods used I calculate that the probable error should be about .1 per cent. of the absolute value, while the probable error found is .09 per cent. This shows that the law holds with milk within the limits of error of the methods of analysis.

In Series 1 I have divided the estimations into four periods—*i.e.*, November to

January, February to April, May to July, August to October—and I find the average error in each of these periods to be as follows :

1893	1st period	—
	2nd „	—·06
	3rd „	+·05
	4th „	+·02

It is remarkable that, in the previous year, when the method was not quite the same and the milk scale was used, that the differences were :

1892	1st period	—
	2nd „	—·04
	3rd „	+·07
	4th „	+·02

Whether the fact that fats calculate low in the spring and high in the summer be due to a difference in the composition of the milk, or some unknown external conditions, cannot be decided ; as practically the same differences have been observed for two successive years, it is probable that this is not a purely accidental occurrence, and it may reasonably be expected to occur again.

There is some evidence that it occurred in 1889, as I then calculated a formula $T = 263 \frac{G}{D} + 1.17 F$, using methods but little different from the present one, which gives results differing about ·07 to ·08 from the present one ; and the work on which this was based was all done in the spring—i.e., in the second period.

In 135 samples of genuine milk the ash has been determined, and the ratio to 100 parts of solids-not-fat calculated.

Ratio of Ash to 100 Solids-not-fat.

Solids-not-fat	Nc. of Samples.	Limits.	Average.
9.4	1		8.5
9.3	1		8.1
9.2	4	8.0—8.4	8.2
9.1	12	7.9—8.5	8.2
9.0	23	7.8—8.4	8.15
8.9	27	7.9—8.5	8.2
8.8	21	8.0—8.4	8.2
8.7	8	7.9—8.8	8.3
8.6	4	8.0—8.6	8.3
8.5	7	8.1—8.7	8.4
8.4	7	8.3—8.7	8.5
8.3	5	8.5—8.9	8.7
8.2	6	8.6—9.0	8.9
8.1	6	8.8—9.4	9.0
8.0	2	8.8—9.3	9.0
7.7	1		9.1

In all these cases the ash was determined with the utmost care, frequently in muffle, but never at a temperature above a very dull red.

A considerable proportion of these have been found with an alkaline reaction to both turmeric and litmus paper, and phenolphthalein; in some the alkalinity has been determined, and a maximum amount of .025 per cent., calculated as Na_2CO_3 , has been found; in some samples a slightly fuller examination has been made.

No.	Percentages on Insoluble Ash.						
	Soluble Ash.	Insoluble Ash.	Na_2CO_3 .	P_2O_5 .	CaO.	P_2O_5 .	CaO.
1.	.26	.54	.025				
2.	.24	.53	.010	.228	.166	43.0	31.3
3.	.28	.48	.020	.205	.152	42.5	31.7
4.	.23	.55	.012	.226	.132	41.1	24.0
	.23	.54			.125		23.1

The soluble ash contained only the merest traces of phosphates, less than .005 per cent. P_2O_5 ; it consisted of chlorides of the alkalis, and the carbonates to which the alkalinity was due; the insoluble ash consisted of a double phosphate of the formula $(\text{Ca}, \text{Mg}) (\text{K}, \text{Na}) \text{PO}_4$, analogous to the carbonates, sulphates and borates of calcium, and the alkali metals. The figures calculated for a calcium potassium phosphate are: CaO 32.2 and P_2O_5 40.5, and for a calcium sodium phosphate, CaO 35.4 and P_2O_5 44.9.

In Nos. 2 and 3 the amount of magnesium was very small and was not estimated; in No. 4 the magnesium estimation was lost, but it was seen to be much larger than in the others.

It is well known that the ash of milk does not represent the salts present therein, but includes phosphoric acid, produced by the oxidation of the phosphorus of the casein, which displaces the carbonic acid formed on igniting the organic salts of milk. A very important paper by Söldner (*Landw. Versuchstat.* xxxv. 351), which has not yet been properly appreciated, has greatly elucidated our knowledge on the subject. He shows that the phosphates present in milk are to a certain extent acid phosphates, and to these in part is the acidity of milk (to phenolphthalein) due. Milk when fresh, and in many cases for some time after, reacts alkaline to litmus. Among the salts the presence of which is established in milk, and which react alkaline to litmus, are citrates and phosphates of the formula M_2HPO_4 ; the practice of calculating the acidity of milk to phenolphthalein as lactic acid is not, therefore, correct; and, indeed, milk freshly drawn has an acidity (calculated as lactic acid) of .1 per cent., or more.

With reference to the presence of nitric acid in milk, when a cow has been dosed with small proportions of nitrates, I thought it desirable to definitely ascertain if the use of water containing nitrates would produce the same effect. After a considerable amount of trouble, I succeeded in obtaining samples of milk yielded by cows accustomed to drink water containing 18.0 parts per 100,000 of nitric acid (as N_2O_5); these samples gave a strong diphenylamine reaction. My previous failure to find nitric acid is due to the fact that it is a *sine qua non* that the water on all the farms supplying the company shall be unpolluted, and therefore contain nitric acid in

only small amount, and that this provision is enforced is shown by the large number (91) of water samples analyzed.

A few experiments were made as to the loss of water experienced on keeping butter samples. A quarter-of-a-pound sample was taken immediately after churning and analyzed at once, the sample was then kept under various conditions and reanalyzed.

As examples I may quote :

Fresh samples,	14.64 %	of water ;	after two days in an open basin,	12.12 %
" "	14.87	" "	" " ten days wrapped in single paper,	13.99 %
" "	13.10	" "	" " " " double "	11.93 %

With a view of studying the connection between the quality of milk yielded by cows and external conditions, I have noted this year the temperature of the air, and I find that it is a general rule that sudden rise of temperature causes a milk of poorer quality to be produced, while a sudden fall has the opposite effect ; should, however, the temperature continue low or high, the quality of the milk gradually returns to its normal level.

There seems to be some evidence that the quality of milk is improved by changing the food from time to time, but that the effect on the quality of the milk is not permanent. The fact that a change is made in the food seems to have more effect than the particular food given ; the evidence on this point is, however, by no means conclusive.

It is usually accepted as a fact that cream contains a larger proportion of solids-not-fat to water than the milk from which it was derived ; and the increase of solids-not-fat is said to be due to the fat globules taking up a larger amount of proteids than the proportion deduced from the ratio of proteids to water in milk. This has been used as an argument as to the presence of an albuminous membrane round the fat globules. I have, however, been led to doubt that this is the case, and have obtained results which point to the ratio of solids-not-fat to water, being the same in cream as in the milk from which it is derived.

The method of analysis which I adopt has been previously described by Dr. Vieth (*ANALYST*, xvi. 1), but as it will be necessary to discuss one or two points, I describe it again in more detail. The sample for analysis is well mixed, and about 5 grammes are weighed into an eight-ounce, flat-bottomed, conical flask ; this is placed in an air-bath at 100°, and is shaken slightly at intervals of fifteen minutes till it is apparently dry, which takes about one hour ; it is then placed on its side, so that the fat shall run away from the solids-not-fat, and left in the air-bath for about four hours. I find that drying beyond this time does not decrease the weight, but rather increases it. A second portion, of 3 to 4 grammes, is placed in a platinum basin, and this is left in the air-bath for five hours, when the minimum weight is obtained. The two results have only differed by more than .1 per cent. in two cases out of forty—viz., .11 per cent. and .25 per cent. The sample in the basin is used for the ash determination ; the flask is partially filled with ether (about 30 c.c.), and this is gently boiled, and allowed to stand for fifteen minutes and decanted, a second quantity of ether is then poured in, and the flask corked and allowed to stand till the following day, during which about six more washings with ether are performed. It

is then allowed to stand again for a night, and a further two or three washings are given; the solids-not-fat are then dried to constancy (about four hours) and weighed. In a few cases the ether has been evaporated and the fat weighed; the weights of the fat and the solids-not-fat do not differ by more than a milligramme or two from the weight of the total solids. In some cases the solids-not-fat have been ground up with a small pestle in the flask, and the ether filtered through a small tared filter; but I do not find that this method is much better than the other. In some cases the fat has been estimated by the Adams or Werner-Schmid methods, and the fat estimated this way and the solids-not-fat add up slightly below the total solids.

The reason for this is not far to seek. I heated in the same bath about 2 grammes of anhydrous butter fat (*i.e.*, shaken with a considerable proportion of calcium chloride at about 50° C. and filtered); the heating was continued for six days, but I need only quote one or two results here:

Time of Heating.		Percentage on Original.		
1½	hours	100.17
16½	"	100.73
20½	"	100.81

As cream contains, say, 50 per cent. of fat, the increase on heating four to five hours will not be quite negligible, and may be estimated at .1 to .2 per cent. on the weight of the cream; and therefore the solids-not-fat determined by the difference between total solids estimated by drying for a long period, and fat estimated by methods in which the drying at 100° C. does not exceed fifteen to twenty minutes, will be increased by this amount.

In the method that I have adopted, there is a possibility that the extraction is not quite complete; and, on the other hand, though the greatest care was exercised in decanting the ether, it being done over a black surface, so that any solid particles going away with the ether might be seen, there is a possibility of loss in this way. I was, however, not able to observe any deposit in the decanted ether.

In the following analysis, the solids-not-fat have been calculated on the assumption that the ratio of the solids-not-fat to water was 10.2 to 100, which I have found to be the mean proportion in the milk which is generally used for the production of cream, except in No. 2, which was prepared by the shallow setting system, and the milk previously submitted to analysis, the ratio being 10.0 to 100; all the other creams were separated:

No.	Total Solids.	Solids-not-Fat.	S. n. F. Calculated.	Difference.
1.	32.50	6.83	6.90	+ .07
2.	37.59	6.14	6.24	+ .10
3.	50.92	5.02	5.01	- .01
4.	55.05	4.65	4.59	- .06
5.	55.18	4.77	4.57	- .20
6.	55.97	4.47	4.49	+ .02
7.	56.37	4.40	4.45	+ .05
8.	57.99	4.17	4.28	+ .09
Average Difference				+ .01

In one cream, No. 1, a proteid estimation was made, and 2.60 per cent. was found, or 38.1 per cent. on the solids-not-fat. The mean proportion of proteids to the solids-not-fat in milk is, according to my estimate, 38.6 per cent.

THE ANALYST.

The ash has also been determined, and the results compared with that calculated on the assumption that it is one-twelfth of the calculated solids-not-fat :

No.	Ash.	Ash Calculated.	Difference.
1. ...	·57	·57	—
2. ...	·52	·52	—
3. ...	·42	·42	—
4. ...	·38	·38	—
5. ...	·39	·38	— ·01
6. ...	·38	·37	— ·01
7. ...	·38	·37	— ·01
8. ...	·41	·36	— ·05

Except perhaps in the last case, the agreement is everything that can be desired.

As lack of time has prevented my making as extensive a series of determinations as might be desired, I cannot say that the statement that cream contains a higher proportion of solids-not-fat than milk is incorrect, but the whole of the evidence that I have obtained points in the direction contrary to that generally assumed. It seems probable that this statement arose from the fact that solids-not-fat determinations were made by difference, the total solids being too high, from an increase of weight of the fat on drying, and the fat possibly too low, owing to a method which gives an incomplete extraction being used (*e.g.*, Soxhlet's method), which I have shown, and Vieth has confirmed my observation, to give too low results. Cf. ANALYST, xiv. 123 ; and xvi. 203.

I have dwelt at length on this subject, as, were it true that cream contained a higher proportion of proteids, a field of investigation would be open to see if the corresponding deficiency of proteids might be utilized in the detection of separated milk as an adulterant ; but I have now little hope of such being the case, and indeed, in a few experiments, I have found no deficiency of solids-not-fat or proteids in separated milk.

An analysis of the slime found on the inside of the drum of a cream separator has been made ; its composition was :

Total solids...	...	33·76	
Fat	·50	
Milk-sugar ...	(about)	·50	
Casein (or analogous body)	„	22·00	
Ash	3·01	
	Soluble ash ...	·166	containing Cl ... ·008
	Insoluble ash...	2·844	„ Silica ... ·171
			Fe ₂ O ₃ .Al ₂ O ₃ ... ·012
			CaO ... ·654 = ·675 eq.
			MgO ... ·225 = ·325 eq.
			Alkalies ... ·559
			P ₂ O ₅ ... 1·233 = 1·506 eq.

The portion returned as casein was that part soluble in dilute alkalies and precipitated by acids. The “silica” was the portion insoluble in hydrochloric acid on evaporation. It is seen that the insoluble ash (minus silica, iron oxide, etc.) has the same general composition as the insoluble ash of milk (see *ante*)—*i.e.*, (Ca, Mg) (Na, K) PO₄.

There are 1.506 equivalents of P_2O_5 (1 eq. = $\frac{7.1}{3}$) for each equivalent of CaO and MgO present (1 eq. CaO = $\frac{5.6}{2}$, 1 eq. MgO = $\frac{4.0}{2}$), and this fact alone furnishes a strong argument that this slime is not (though it contains) the dirt in the milk, and that it is a true milk product.

The ash was absolutely neutral (litmus and phenolphthalein) in reaction.

DISCUSSION.

Mr. Otto Hehner said that Mr. Richmond had extracted much interesting matter from a subject which had been very often discussed before. Sooner or later the Law Courts would have to decide the question as to whether butter was a perishable article or not. For the purposes of analysis, butter could hardly be called a perishable article, as it could be reliably analysed when many months old. Much of the confusion that had arisen from the conflicting statements of different observers—some of whom said that the percentage of insoluble fatty acids would not materially alter on keeping the samples, others that they decreased—was due to the fact that some analysts kept the butter entire, others the butter-fat. He was surprised that Mr. Richmond had not found any carbonates in his milk-ashes. He would rather have expected that there would have been a notable quantity. In sweetened condensed milks a deposit was generally observed, which consisted largely of calcium citrate, and which yielded on incineration calcium carbonate. He [Mr. Hehner] would like Mr. Richmond to state the lowest solids-not-fat which he had obtained.

Mr. Richmond, in reply, said that he could hardly give Mr. Hehner the lowest solids-not-fat, because the samples, which he believed were perfectly genuine, were not samples which were strictly authenticated in the legal sense. All the samples low in solids-not-fat were, however, abnormal in composition. With regard to the absence of carbon dioxide in the ash of separator-slime, it was not exactly the same thing as in the case of the deposit found in condensed milk, because it should be remembered that the condensed milk had been heated to a certain degree, and calcium citrate was known to be more insoluble in hot water than cold. In the ash of milk itself there was a small quantity of carbonic acid in a considerable number of cases. He had mentioned that in the soluble ash there was sometimes carbonic acid to the extent of 0.025 per cent. $NaCO_3$, and beyond that there was also a little carbonate in the insoluble ash. With respect to Mr. Hehner's remarks as to how difficult it was to wash milk-ashes, he thought a very possible explanation was that the alkali existed there as calcium-sodium-carbonate. He wished to mention that although the paper was in his name, the bulk of the analytical figures were the work of his assistant, Mr. Boseley.

[NOTE.—In a recent number of the *Comptes Rendus* there is a paper on the preparation of double carbonates of potassium and sodium with calcium, barium, and strontium, and they are described as well-defined crystalline compounds of limited solubility.—H. D. R.]

NOTE ON THE DETECTION OF COTTON-SEED OIL IN LARD.

By E. J. BEVAN.

Read at the Meeting, February 7, 1894.

SOME time ago I had occasion to examine a sample of lard for cotton-seed oil. This lard, which I knew to be genuine, gave the silver reaction. On finding this, the first thing I did was to make a new silver solution, but I found that the sample gave the reaction with all the solutions tried. It so happened that the bottle containing this lard had been long exposed to the air and the fumes of the laboratory. This gave me a clue to the matter. By scraping off the top portion, and taking a part of what lay underneath to which the air had not penetrated, I obtained no silver reaction whatever. I then took a sample of bladder-lard, and I observed the same phenomena. The portion immediately under the skin gave the silver reaction readily, whereas a portion taken right inside the mass in the bladder gave no reaction whatever. The fact that the lard underneath the skin gave the reaction showed, of course, that it was not due to the presence of any mechanically-deposited dirt. Then I tried exposing the lard in a still room in flat dishes. I found that after about a week's exposure the lard gave the silver reaction quite strongly. I can come to no other conclusion than that the effect observed was due to oxidation. I should say that I also exposed the lard to the fumes of various substances without afterwards obtaining any silver reaction whatever. I passed air for two or three days through some melted lard, and I found that with the lard so treated the reaction was intense. I estimated the amount of iodine absorption, the free acid, and one or two other things, and found no differences whatever, so that evidently the substance which is formed must be very minute in amount, but there is quite enough to mislead one, and give one the impression that cotton-oil is really present.

I notice that in the January number of the journal of the Chemical Society there appears an abstract of a "Note on the Reducing Action of Rancid Fat." It is stated by the author of that note that butter which has become rancid gives the cotton-seed oil reaction in a marked manner, and he also goes on to say that he considers the reaction to be due to the presence of sulphur compounds in lard, like those in certain vegetable oils. I do not think that this is true. It is known that lards do not contain sulphur compound. I cannot see that the effect is due to rancidity.

Mr. Chattaway said that it seemed to him strange that if the presence of free acid was in any way the cause of what Mr. Bevan had demonstrated, one should not get the reaction with the fatty acids themselves. He had very frequently tested fatty acids, and obtained no silver reaction.

Mr. Otto Hehner said that rancidity was not solely due to liberation of fatty acids, but was largely attributable to oxidation. Rancid fats, when saponified with alcoholic potash, gave a more or less yellow solution, similar to that produced when alkalies acted upon aldehydes; his impression was that rancidity was accompanied by the formation of aldehydic bodies. Mr. Mariani had pointed out that rancidity might cause butter-fat to yield a reaction with silver nitrate similar to the Becchi reaction. He (Mr. Hehner) advised members not to put too much trust in the cotton

seed oil reaction when applied to butter, as he had found that when cows were fed with large quantities of cotton-seed cake, the butter obtained from the milk of cows so fed would not unfrequently give the reaction for cotton-seed oil.

He would like to elicit from members some information concerning lard. A few years ago, when cotton-seed oil was frequently present in lard, the iodine absorption was largely relied upon in lard-testing. Lately, cotton-seed oil was but very rarely met with in lard, yet the iodine absorption had, if anything, increased, although beef-stearin, which had a very low iodine number, was often present. Hence the presence of some oil other than cotton-seed or lard oil must be suspected.

Mr. Cassal drew Mr. Bevan's attention to the fact that according to the abstract of Mr. Mariani's paper in the journal of the Chemical Society he appeared to have stated that he had shown lard and animal fats to contain sulphur compounds, and to have based that statement upon his having obtained the silver reaction with these fats, and upon his having found the deposit to consist partly of sulphide of silver.

Mr. Richmond, with respect to the presence of sulphur in the fat of animals pointed out that when he was in Egypt he examined the milk of the gamoose, and in the fat there was 0.05 per cent. of sulphur, and it gave, after saponification, a very strong reaction with both silver and lead paper. He thought it possible that, as sulphur was present in the fat of one animal, it might also be found in that of others.

Mr. Bevan said that he had only read the passage casually, but it seemed to him that there was not sufficient evidence upon which Mariani could base his statement.

CIDER VINEGAR.

By GEORGE S. COX.

IN connection with the enforcement of the Wisconsin vinegar laws (which require the presence of 4 per cent. of acetic acid and of 2 per cent. of solids in cider vinegar), the writer has spent some little time in the examination of these lines. In *THE ANALYST* for 1891 (p. 41) Mr. G. Embrey presents a paper devoted mainly to the consideration of the percentage of ash in cider. Upon reviewing some of the work done in the State laboratory the past summer I find some figures that may be of interest to your readers, taken in connection with Mr. Embrey's paper.

Mr. Embrey finds the ash to range from 0.25 per cent. to 0.35 per cent., a figure lower than 0.25 per cent. carrying the assumption of added water; but in regard to a figure higher than 0.35 per cent. no statement is made. On p. 42 is a criticism of R. Kayser's figures as quoted by Allen in his "Commercial Organic Analysis." Kayser gives the total solids of must as 16.25 per cent. and of cider as 2.36 per cent. The sugar of the former is given as 12.5 per cent. and of the latter as 0.75 per cent. Mr. Embrey says: "There is evidently some mistake here, as the total solid matter of cider is given as 2.36 per cent.—probably 12.36 per cent." The fact is here overlooked that the must has a high sugar content that has been removed from the cider by fermentation. With the trade in this country, the product of the cider mill and press is called "juice" before fermentation and "cider" after, and from the context of this

paper this same practice seems to obtain in England. In accordance with this, Kayser's figures are certainly more nearly correct than 12.36 per cent.

The table here given requires some additional explanation in addition to the remarks. 601, 602, 603, and 607 were samples of vinegar that had been made by the quick process in Gould generators. The other vinegars were made by storing the juice in barrels in a dark warehouse at a moderately warm temperature, and the dates mentioned refer to the time at which barrels were placed in storage. 626, 629, 630, and 631 were samples of juice pressed in 1893 (November), and examined within a few days after their manufacture. 608 to 620 inclusive the writer withdrew from the barrels in which it had been stored. The samples of juice were furnished by the manufacturer of the vinegars, and are just as represented.

Sample number.	Article examined.	Percentage of acid as acetic.	Percentage of total solids.	Percentage of ash to total amount.	Percentage of ash to total solids.	Remarks.
601	Vinegar	4.47	2.4	0.25	10.41	
602		4.92	3.1	0.25	8.06	
603		5.45	4.0	0.26	6.50	
604		5.79	1.64	0.31	18.90	J. Put away in brls. in Oct., 1891.
605		7.56	1.54	0.34	22.08	J. Put away in brls. but a few weeks earlier.
606		6.09	2.29	0.36	15.72	J. Same date. Made from snow apples.
607		5.29	2.43	0.259	10.65	
608		7.74	1.77	0.435	24.57	J. Oct., 1891. Apples.
609		6.19	2.17	0.40	18.43	J. Oct., 1891. Port wine brl.
610		7.29	1.34	0.52	38.80	Ditto.
611		8.40	2.66	0.39	14.66	J. Oct., 1891. Charred whisky brl.
612	"	7.86	2.55	0.35	13.72	Ditto.
613		6.19	2.23	0.34	15.24	Ditto.
614		2.91	1.89	0.33	17.46	Put in warm storage. Fall of 1892.
615		3.78	3.17	0.26	8.20	Ditto.
616		7.81	1.43	0.33	23.07	November apples. 1891.
617		5.32	2.74	0.43	16.42	Emptyings of brls. Three years old.
618		2.28	2.09	0.30	14.35	November apples. 1892.
619		5.63	1.55	0.30	19.29	November apples. Larger bung.
620		8.09	2.27	0.40	17.62	Eight years old.
626	Juice		14.83	0.525	3.54	Solids dried twelve hours.
629			13.36	0.286	2.14	New York apples; not very sound.
630	"		11.25	0.331	2.94	Partly fermented salicylic acid present.
631	"		9.48	0.563	5.93	Same as 630, but from a vinegar barrel.
632	Settlings		2.54	0.246	9.68	Settlings from cider brls.

The average ash for the twenty-five samples is 0.3546 per cent.—a trifle higher than Mr. Embrey's maximum figure—and in only one instance (632) does the figure fall below the minimum; and certainly the settlings cannot be taken as a representative sample.

In samples 626, 629, 630, and 631 the solids were obtained after twelve hours drying on a steam bath. The evaporations were made in flat-bottomed platinum dishes, quite shallow and very thin.

The enforcement of this vinegar law has occasioned some very vexatious questions, and the Wisconsin Commission has given some little time to its consideration. Other points of interest present themselves in addition to the ash figures. Several of these vinegars cannot be sold, under existing statutes, as cider vinegar, while 603 could be watered very considerably and pass our State standard. Mr. Embrey's ash figures would prove little, as the ash is practically the same in 601, 602, and 603, yet the ratio maintained between the solids and acid would point to the addition of water. The goods were from the same place, in the same consignment, and were billed as 5 per cent., 4.5 per cent., and 4 per cent. acid strength respectively, and were evidently mixed with water in proportion to approximate the desired acidity. 618 and 619 were from the same run of juice, and stood side by side. They were examined eleven months after pressing, and are interesting as exhibiting the marked effect of a slight variation in the air-vent. 604, when received, contained nearly 4 per cent. by volume of alcohol that had not yet been converted, and the sample, in a half-gallon bottle, together with 605 and 606, was placed in the direct sunlight in a warm room, where it was allowed to stand for several months. The two other samples had little or no unconverted alcohol, and were practically unchanged, while 604 developed a bouquet and flavour incomparably superior to the others. Previously they had been stored in the dark and at a lower temperature, and when received were very similar in taste and odour, but the sunlight and warmth toward the close of the acetic fermentation created a difference that cannot be lightly estimated, from the commercial standpoint, at least.

Experiments upon this line are now in progress, and should anything of interest occur I shall be very glad to give you notes regarding it in a future contribution.

*Wisconsin Dairy and Food Commission,
Madison, Wis., U.S.A.*

NOTE ON G. S. COX'S PAPER ON CIDER-VINEGAR.

By A. H. ALLEN.

Mr. Cox's results are far more suggestive when calculated on the "original solids" of the apple-juice, and many of the anomalies disappear. The ratio of the ash to the residual solids of the vinegar has little meaning or interest, but the proportion it bears to the "original solids" of the juice before fermentation affords much assistance in forming an opinion as to the extent to which dilution has been practised.

The following table shows the "original solids" of Mr. Cox's samples calculated by *Hehner's formula*, and the ash for 100 parts of these solids. In calculating the

original solids of Sample 604 the alcohol present must be taken into consideration. But the correction I have applied is evidently excessive.

Sample Number.			Original Solids, per cent.	Ash on Original Solids, per cent.
601.	...	Vinegar	9.10	2.74
602.	...	"	10.48	2.38
603.	...	"	12.17	2.13
604.	...	"	16.00	1.94
605.	...	"	12.88	2.63
606.	...	"	11.42	3.15
607.	...	"	10.36	2.50
608.	...	"	13.38	3.25
609.	...	"	11.45	3.49
610.	...	"	12.27	4.23
611.	...	"	15.26	2.55
612.	...	"	14.34	2.44
613.	...	"	11.51	2.95
614.	...	"	6.75	4.88
615.	...	"	8.84	2.94
616.	...	"	13.14	2.51
617.	...	"	10.72	4.01
618.	...	"	5.51	5.44
619.	...	"	9.99	3.00
620.	...	"	14.40	2.78
626.	...	Juice	14.83	3.54
629.	...	"	13.36	2.14
630.	...	"	11.25	2.94
631.	...	"	9.48	5.93

THE DETERMINATION OF IMPURITIES IN COMMERCIAL COPPER.

By BERTRAM BLOUNT.

THE method given by Professor Hampe (*Chem. Zeit.*, 1893, xvi, 1691, see abstract on next page) depending upon the precipitation of the copper as cuprous thiocyanate and the determination of the metals present as impurities in the filtrate, has been in constant use in my laboratory for some years past, having been devised by me without knowledge of its previous employment.

The principle of the process is identical with that adopted by Professor Hampe, but there are a few differences in detail which add somewhat to its convenience. In most cases 10 grammes of the copper to be analysed is sufficient to yield ponderable quantities of the impurities to be determined, although of course this quantity may be increased if necessary. In fact, the great merit of the method consists in the fact that large quantities of material may be used without the trouble of handling unwieldy precipitates, as when once the copper has been precipitated as thiocyanate nothing further has to be done to it in the way of manipulation. When first working the process, I was accustomed to use 10 grammes of the copper and wash the resulting thiocyanate. This plan was speedily abandoned in favour of that mentioned by Professor Hampe, of drawing off a definite portion of the supernatant liquor and allowing for the volume occupied by the precipitate. In order to do this, it was

necessary to ascertain the specific gravity of cuprous thiocyanate. This constant was not given in any book which I consulted, and accordingly was determined on a sample prepared in the course of one of the previous analyses. The figure thus found for the specific gravity of cuprous thiocyanate at 15° C. compared with water at the same temperature, was 2.846, somewhat lower than that found by Professor Hampe (2.999). The reason for this discrepancy may be that the precipitation was conducted under different conditions which had an influence on the density of the salt. Taking the figure for the specific gravity of cuprous thiocyanate given above, a simple calculation showed that if 13.215 grammes of copper were taken, dissolved, precipitated as thiocyanate, and the solution together with the precipitate made up to 1 litre, on drawing off 750 c.c. the impurities corresponding to 10 grammes of copper would be obtained. By using this plan, all need for calculation of the results of the analysis is avoided.

Another point in which my process differs from that of Professor Hampe is that the copper is dissolved in aqua regia instead of nitric and sulphuric acids. By the use of this solvent, and the subsequent removal of the excess of nitric acid, I obtain a clear solution with all ordinary samples of copper, and avoid the necessity for making a separate analysis of the insoluble residue, a result which is, I venture to think, a distinct advantage. Thus all the common impurities of commercial copper, with the exception of gold, silver, sulphur, and oxygen, can be determined in one and the same solution, in the absence, moreover, of all but traces of copper, making the analysis a vastly simpler task than by the older methods.

There is only one other matter that deserves mention. Although arsenic can be determined in the solution from the cuprous thiocyanate, I have found it more expeditious, easy, and accurate to estimate it on a separate portion by distillation with ferric chloride and hydrochloric acid, as it is thus finally obtained as pure arsenious sulphide, and can be conveniently weighed in this form. There is only one drawback to this process, and that is the difficulty of obtaining reagents free from arsenic, making it necessary for the chemist to prepare them himself.

BROADWAY, WESTMINSTER, S.W.

The Determination of Foreign Metals in Commercial Copper. W. Hampe. (*Chem. Zeit.*, 1893, xvii. 1691-1692.)—The following is the method adopted by the author: 25 grammes of the sample are dissolved in a mixture of 200 c.c. of water, 100 c.c. of pure concentrated sulphuric acid, and 45–46 c.c. of nitric acid of sp. gr. 1.21. The quantity of the last-named reagent is reckoned so as to afford a small excess over that necessary for the oxidation of the quantity of copper taken, while the amount of sulphuric acid represents a considerable surplus, in order to prevent the separation of basic salts of bismuth and antimony when the solution is subsequently diluted. When the whole of the copper is dissolved, the solution is diluted with 200 c.c. of water to prevent the formation of crystals of copper sulphate. The resulting liquid is generally clear, but it may be turbid from the separation of insoluble antimonates of copper and bismuth, which must in that case be filtered off and examined separately. The original solution, or the clear filtrate, as the case may

be, is warmed to 40° C., and treated with sulphur dioxide in a rapid stream to decompose the remainder of the nitric acid, the reduction being complete in about half an hour provided the temperature specified, which is the most favourable for the reaction, be observed. The solution, which should smell of sulphur dioxide, may be turbid from the presence of metallic silver precipitated by the reducing agent. Should it be desired to determine the silver in the wet way, the precipitation of traces not reduced by the sulphur dioxide is effected by the addition of a few drops of hydrochloric acid and the mixed precipitate of silver and silver chloride filtered off, converted completely into chloride and weighed in the usual manner. If, on the other hand, a dry assay for silver is to be made, the turbidity due to the separation of metallic silver may be disregarded, and the main body of liquid, together with the trace of metallic silver, is transfixed to a two-litre flask and precipitated with pure potassium thiocyanate, a rapid stream of sulphur dioxide being meanwhile maintained. A slight deficiency of potassium thiocyanate is used, so that a small fraction of the copper may remain in solution. The solution of potassium thiocyanate is of such strength that about 500 c.c. are required to precipitate 25 grammes of copper. The total bulk is then made up to two litres, the precipitate allowed to subside and a known volume of the supernatant liquor filtered off. 1800 c.c. is a convenient amount to take. The excess of sulphur dioxide is driven off by evaporation, and the foreign metals originally present in the copper, such as antimony, arsenic, bismuth, tin, iron and nickel, are separated and determined by the customary analytical methods. In making the calculations of the analysis it is necessary to correct for the volume of the cuprous thiocyanate in order to ascertain with what fraction of the 25 grammes of copper originally taken the quantity of liquid drawn off after precipitation corresponds. This involves a knowledge of the specific gravity of cuprous thiocyanate. The author has determined this value, and finds it to be 2.999, so that the volume occupied by the cuprous thiocyanate from 25 grammes of copper is 15.98 c.c. The total bulk of liquid in the two-litre flask may therefore be taken as 1984 c.c., and the relation between this number and that of the liquid drawn off, viz., 1800 c.c., determines upon what fraction of the 25 grammes of copper the estimation of foreign metals has been effected. Test analyses of pure copper, to the solution of which known quantities of impurities had been added, prove the accuracy of the method.

B. B.

The Separation and Volumetric Estimation of Lead. Lindeman and Mottou. (*Bull. Soc. Chim.*, 1893, x, 812, through *Chem. Zeit.*).—The authors have found that chloride of lime is capable of acting on native sulphides, converting their metals into oxides or peroxides and their sulphur into sulphate. The oxidation appears to be complete, and thus can be utilised for the estimation both of the metal and sulphur. In the case of galena the lead is obtained as peroxide, and can be estimated iodometrically. The analysis is performed by rubbing down 0.5 to 1.0 gramme of the sample in an agate mortar with a few drops of a solution of chloride of lime, and finally triturating the product with 30 to 40 c.c. of the same reagent. A little hydrochloric acid is then added, and the whole warmed until chlorine ceases to be evolved. The solution is diluted, and the lead peroxide washed by decantation, bringing as little as possible upon the filter, and treated with potassium iodide and hydrochloric acid; the liberated iodine is titrated with hyposulphite. The presence

of either iron or copper interferes, as each is capable of liberating iodine and is therefore reckoned as lead. When iron alone is present it is sufficient to precipitate the lead as sulphide with sulphuretted hydrogen in acid solution, and treat the sulphide as if it were the original ore. When copper has to be removed, the sulphide is precipitated as described above, and washed with potassium cyanide, which dissolves the cupric sulphide and leaves the lead sulphide to be dealt with as before.

B. B.

The Determination of Phosphoric Acid by the Titration of the Yellow Precipitate with Standard Alkali. (*Jour. Franklin Inst.*, 1893, cxxxvi., 362-376.)

—The author shows that 23 molecules of sodium carbonate are required to neutralize the molybdic acid in every molecule of the yellow precipitate when the composition of this is $6\text{NH}_4\cdot\text{P}_2\text{O}_8\cdot 24\text{MoO}_3$. The best standard alkali for the purpose is a solution of caustic potash, of which 1 c.c. is equivalent to 1 milligramme of P_2O_5 in the yellow precipitate. Such a solution neutralizes normal acid in the proportion of 100 : 32.65 volumes, and may be made from potash which has been freed from carbonate by means of baryta-water, on this basis; or it may be standardized by titration of the yellow precipitate obtained from pure sodium phosphate, treated in the manner described below.

The ammonium molybdate solution is prepared by dissolving 90 grammes of the salt in somewhat less than a litre of water, allowing the solution to settle, and decanting the clear liquor into a litre flask. The small quantity of insoluble molybdic acid always present is dissolved in a little ammonia, and added to the main solution. If the molybdate contains a trace of P_2O_5 , a few decigrammes of magnesium sulphate, and ammonia to faint alkalinity, are added. The whole is then made up to one litre. Each c.c. precipitates 3 milligrammes of P_2O_5 .

The standard acid must be equivalent to the alkali, and may be made by adding 326.5 c.c. of normal acid to one litre.

The analysis is conducted as follows: One gramme of phosphate rock, or 2 or 3 grammes of fertilizer, are dissolved in nitric acid, and, without evaporating to dryness, diluted to 250 c.c. The solution need not be filtered, inasmuch as the volume of the insoluble matter seldom amounts to 0.05 c.c. Twenty-five c.c. of the solution are delivered into a four-ounce beaker and neutralized with ammonia—until a precipitate just begins to form—and then treated with 5 c.c. of nitric acid of 1.4 sp. gr. Ten c.c. of a saturated solution of ammonium nitrate are added, and the solution diluted to a volume of 50-75 c.c. It is then brought to a full boil, removed from the lamp, and 5 c.c. of the aqueous solution of ammonium molybdate added. This is followed by a second and a third 5 c.c. if necessary, the precipitate is allowed to settle, and filtered at once. It is washed thoroughly by decantation and on the filter. The filter and precipitate are transferred bodily to the beaker. Standard alkali is then run in, and at least 0.5 c.c. of a phenolphthalein solution (1 per cent.) added, then standard acid until the colour vanishes. Each c.c. of alkali equals one milligramme of P_2O_5 .

A. G. B.

REVIEW.

THE MICRO-ORGANISMS OF FERMENTATION. By ALFRED JÖRGENSEN. London: Lyon. Price 10s.

The morphology and biology of the organisms concerned in the many varieties of fermentation, which occur naturally or are induced artificially, now possesses a wide and diffuse field of literature of its own. In the work before us, the author

has attempted to bring together, within fairly small compass, the salient points of this enormous mass of literature, and to give a general view of the subject in so far as it concerns the great fermentation industries. Working on these lines, he has succeeded in producing an exceedingly readable volume of handy size, which contains matter of the utmost interest to the chemist, biologist, and botanist. In the general arrangement of the work, the methods for the microscopical and biological examination of bacteria and yeast fungi are first described, this being followed by methods for the examination of air and water for such organisms. An account of the more commonly-occurring bacteria and mould fungi comes next. This is followed by a description of that numerous class of bodies—the alcoholic ferments—together with a short *résumé* of the various theories of fermentation, and this naturally forms the most important section of the work. An account is then given of the methods for the preparation of pure cultures of yeast on the principles first enunciated by Dr. Hansen, of the Carlsberg Laboratory. The last chapter is dedicated to a description of the beneficial results which have accrued in actual practice, where the principles, which have been gradually evolved during the last few years from our increased knowledge of the organisms of fermentation, have been applied. A list of the extensive bibliography of the subject concludes the work. The present volume, which is the second edition of the work in the English language, is a translation of the enlarged third German edition, and great credit is due to the translators, Dr. A. K. Miller and Mr. E. A. Lennholm, for the excellent manner in which they have performed their task. The book is well and clearly printed, neatly bound, and contains numerous illustrations. We can strongly recommend it as an indispensable manual to all those who are engaged in any of the industries where fermentation plays an important part.

W. J. S.

CORRESPONDENCE.

To the Editors of THE ANALYST.

SIRS,—I am sorry to see that an allusion to raspberry-jam in my paper on Vinegar, recently read before the Society of Public Analysts, should have been misconstrued by Messrs. Allen and Hehner. My argument was, that where an identical result can be obtained by alternative methods, it is not the duty of the analyst to go behind the result.

As my thoughts have been previously misunderstood, perhaps I had better add that I don't look for the conversion of turnips into raspberries; but if it were possible, I should consider the author of the process a public benefactor rather than a dishonest manufacturer.—Yours faithfully,

Stourport, February 21, 1894.

EDWARD COLLENS.

To the Editors of THE ANALYST.

DEAR SIRS,—A sentence has, through a printer's error, crept into our paper on "The Leffmann-Beam Method," *ANALYST*, this volume, p. 68, lines 13 and 14 from bottom.

It was not in the original paper, but formed portions of a sentence on the wrapper used to keep the paper clean; the sentence being incomplete, was not sense as it stood, and the printer made an effort to form readable English by putting in an "is" where we had written "as." This, though making the sentence read well, entirely altered its meaning, and seemingly commits us to a direct attack on the Babcock method. As will be seen in the previous portion of the paper, we have studiously avoided any reference to the Babcock process, which has not had for its object the elucidation of conditions of the Leffmann-Beam method.—Yours truly,

H. DROOP RICHMOND.

L. K. BOSELEY.

Bayswater, March 28, 1894.

THE ANALYST.

MAY, 1894.

DR. ARTHUR HILL HASSALL.

By the death of Dr. Hassall, which occurred at San Remo, there has been removed from our midst one to whom the public and all Public Analysts owe a debt of gratitude, though the period of his activity, during which he became widely known, lies so remote from the present time, that to many of the younger generation his personality represents little more than a name, and that one which is often pronounced with little respect.

Some months since, Dr. Hassall published an autobiography entitled "The Story of a Busy Life," which, though affording an insight into the life and character of this remarkable man, makes no reference to many matters on which information would have been acceptable; and which, to many who knew Dr. Hassall, would have been quite as interesting as those actually contained in the book. He has left his mark beneficially on a number of divisions of natural science, although he had to work under conditions of poverty and ill health, which would have proved an effectual bar to persons less energetic and mentally less active. Quite early in life he wrote a "History of the British Fresh Water Algæ"; in this many new species were described, and the work is, we believe, considered authoritative on the subject to this day. Soon after followed "The Microscopic Anatomy of the Human Body," which consisted chiefly of well-executed drawings; further, he wrote on "The Urine in Health and Disease," and he was one of the earliest to observe the occurrence of indican in the urine. Later still he founded the Royal National Hospital for Consumption and Diseases of the Chest at Ventnor, a hospital consisting of several blocks, and acknowledged to be a model of the Cottage Hospital type.

But it is mainly in connection with the adulteration of food that Dr. Hassall's claim to public recognition rests. As early as 1850 he applied the microscope to the examination of food, and though this at the present time may seem to be a mere matter of course, and one which requires no ingenuity, yet up to that date it had not occurred to any one to put this simple idea into actual practice. Dr. Hassall showed that it was easy to distinguish between coffee and chicory by means of the microscope at a time when this subject was a matter of Parliamentary investigation; and even after Hassall's discovery, the then Chancellor of the Exchequer, Sir Charles Wood, stated in the House of Commons: "I hold in my hand the report of the most distinguished chemists of the day, who state that neither by chemistry nor in any other way can the mixture of coffee with chicory be detected." This episode led to the institution, or rather the revival, by Mr. Wakley, at that time the editor of *The Lancet*, of the so-called "Analytical Sanitary Commission," which, in reality, was not

a commission at all, as it consisted solely of Dr. Hassall. The object of the so-called commission was to publish reports, partly for the public benefit, but mainly for the owners of *The Lancet*, on articles of food purchased in all the more important English towns, boldly stating the names of the vendors of the various articles, and the results of the examination of the same. These reports, which extended over a period of several years, and included the results of the examination of many thousands of samples of food, caused an immense sensation. They were mainly founded upon the examination under the microscope of those articles in which adulteration could be detected by that instrument; but it was gradually found that to this method of examination that of chemical analysis must be added. As Dr. Hassall had no chemical knowledge beyond that which had survived from his medical student days, he obtained the assistance of Dr. Letheby for that portion of the work. Ultimately the publication of *The Lancet* reports led to Parliamentary action being taken, and this resulted in legislation against the adulteration of food; the reports were subsequently collected by Dr. Hassall and published separately. We do not wish to do more than refer to the quarrel in the public press between Dr. Hassall and Dr. Letheby, both of whom claimed credit for the reports; nor to that between the editor of *The Lancet* and the member who formed the Sanitary Commission. After the publication of these reports, Dr. Hassall became for a time the chemical oracle of the public, and this upon the basis of his microscopic work, which part of his work was in every way excellent. To him public analysts owe most of their knowledge of the microscopic structure of food substances. His activity gave articulate form to the outcry against adulteration; he was truly the father of public analysis. In recognition of his great services he was elected the first vice-president of the Society of Public Analysts, but he never took any active interest in the formation or actual working of the society.

To the end of his life he remained what he essentially was, a microscopist, not a chemist, or an analyst in the modern sense of the word. His merits are none the less for that fact, and his name will be long and gratefully remembered by the members of the analytical profession.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on April 4th at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner took the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed for election as members: Messrs. Edward M. Chaplin, Thompson's Yard, Westgate, Wakefield; B. Henry Gerrans, 47, Aubert Park, Highbury, N.; William Marshall, 15, West Street, Rochdale; and B. H. Mumby, M.D., Portsmouth.

SOME FREQUENTLY-NEGLECTED ERRORS OF ANALYSIS.

BY H. DROOP RICHMOND.

IN many commercial laboratories it is the practice for several experimenters to weigh on one balance and with one set of weights; and in more than one that I have known the weights are left on the pans when the weighings are finished, and the weight-box is left open. In laboratories where such is the practice, the standardizing of weights, and the daily adjustment of the balance to its zero point are also neglected.

I had occasion once to use a set of weights that had been used continually for some years in a busy laboratory, in which the standardizing of the weights was considered superfluous, and took the opportunity of comparing them to see to what extent errors might arise from this cause. The results were:

Nominal Weight.	Real Weight.	Nominal Weight.	Real Weight.
10'	Taken as Standard	·5	·4982
10"	9·9997	·2	·1993
5	4·9990	·1'	·0971
2	1·9991	·1"	·0982
1'	·9987	·05	·0485
1"	·9989	·02	·0191
1'''	1·0008	·01'	·0092
		·01"	·0089

As an example of the error which may occur, I quote the analysis of a commercial sulphate of copper. This is an actual example obtained with other weights; I have supposed, for purposes of illustration, that the above weights were used:

Tare of watch glass	4·3370	
Watch glass and sample	5·3387	Supposing 1''' had been added the real weight is
			1·0017	1·0025
Tare of Platinum	13·0032	
Platinum and Copper	13·2373	Supposing ·2, ·02, and ·01' had been added the real weight is
			·2841	·2317
			Apparent.	Real.
Percentage: Cu	23·37	23·11
CuSO ₄ ·5OH ₂	91·83	90·82

I have here chosen a striking case for purposes of illustration, but it is one that may occur in many laboratories, and may lie in opposite directions with different analysts; this possibly is a partial explanation of the differences between chemists in commercial analysis. I have found that two or three hours is sufficient for the comparison of a set of weights, and would recommend Gauss' method of reversal (*cf.* Miller, *Phil. Trans.*, 1856, iii.) as the most useful method of determining them.

In the majority of balances that I have used the zero point has varied with the load; thus in a long-beam Oertling I found:

Load in each Pan.	Zero Point in Milligrammes.
none	0.0
1 gramme.	+ 0.3
2 "	+ 0.6
5 "	+ 1.0
10 "	+ 1.2
20 "	+ 1.7
50 "	+ 3.6

By the ordinary method of weighing there would be .03 per cent. difference in the apparent value of a 1 gramme weight, according as it were weighed with no other load or with a load of 50 grammes. This would be of no practical importance in ordinary work, but it illustrates the absurdity of stating results to the third place of decimals per cent., which I have seen on commercial certificates of analysis.

Neglect of correction of weighings to a vacuum is invariably practised in commercial analysis, yet it may affect the fourth place of decimals in density determinations of strong sulphuric acid, and may cause errors of nearly 0.1 per cent. in normal solutions, if barium sulphate, or silver chloride, or bromide be weighed.

That graduated instruments are frequently incorrect is universally known, but in many laboratories they are used as sold without being checked.

It is a less well-known fact, though a committee of the British Association has reported on this, that blowing out pipettes is less accurate than simply allowing them to drain, and then touching the end of the pipette against the side of the vessel in which it has been allowed to drain. It must also be remembered that a pipette which delivers 10 c.c. of water will not deliver the same volume of sulphuric acid or milk or of any fluid of differing viscosity; two 10-c.c. pipettes, each correct for water, may not deliver the same volume of sulphuric acid, and errors in oil analysis by the Maumené method may thus arise.

Turning to the errors of standard solutions, the expansion by heat is a cause of error; thus, with normal sulphuric acid, calling the value 15° 100, the following values at various temperatures are found:

Temperature.	Value.
10°	100.12
15°	100.00
20°	99.85
25°	99.69
30°	99.52

As the extremes in the table may be taken to represent the temperatures attained in a laboratory in summer and winter, it follows that a difference of 0.6 per cent. may be due to the neglect of expansion of a standard solution.

Another error in alkalimetric titrations may occur from the standardization of baryta solution on normal sulphuric acid; I have already referred to this (ANALYST xvi., 166), but as since then Richards has redetermined the atomic weight of barium, I have corrected my figures for this.

Thus the standard acid used contained 5.08° per cent. of H_2SO_4 (using 137.5 as the atomic weight of barium and correcting the weighings to a vacuum standard) instead of 5.104 per cent. The percentage deduced from Pickering's tables was found to be, on fully correcting the density determinations, 5.093 per cent., the corrected densities being

at	$\frac{1}{4}^\circ$	1.00381
and	$\frac{20}{4}^\circ$	1.03218.

The following determinations of strength of the baryta solution were found at 25° :

14.14 c.c. gave	.1705 gra. BaSO_4	=	.1041	Normal
39.48 " "	.4790 " "	=	.1039	"
29.48 " "	.3588 " "	=	.1042	"
Titrated with $\frac{N}{10}$ Hydrochloric Acid		=	.1040	"
Titrated with Oxalic Acid		=	.1042	"
Mean1041	"

Titrated with the Sulphuric Acid referred to above it was

.10301	Normal
.10315	"

Mean10308	"
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It is seen that titration with sulphuric acid shows the baryta solution to be 1 per cent weaker than it really is, and this is no doubt due to the carrying down of barium hydroxide by the barium sulphate.

I may mention also that a standard silver solution has the same property, and that in Mohr's method of chlorine estimation, the value of the silver solution as estimated from the silver contained differs from that obtained by titrating pure sodium chloride by fully 1 per cent. (see ANALYST, xiv., 229). The work of Stas has explained this. The influence of carbon dioxide in solutions of acid has been lately discussed.

With one more fact which is not widely recognised, I bring this somewhat rambling paper to a close, Platinum is permeable at a red-heat by the reducing gases of a Bunsen flame, and precipitates such as copper oxide and manganese dioxide may be somewhat reduced, and the copper oxide may contain less oxygen than CuO , and the manganese may be weighed as a lower oxide than Mn_3O_4 . This may be avoided by the use of the muffle.

DISCUSSION.

Mr. Hehner thought the society would feel obliged to Mr. Richmond for calling attention to facts with which all analysts were supposed to be acquainted. It was highly desirable to test weights occasionally.

Dr. Walter J. Sykes mentioned that properly graduated instruments could be obtained from some firms on the Continent at prices a little higher than those charged for the ordinary ones in England.

Mr. Arthur Ling said it was desirable that the measuring-vessels used for volumetric analysis by one observer, or in any given laboratory, should be verified by means of one set of weights, because sets of weights often differed in standard. With regard to the measurement of liquids by pipettes, one of the best plans was that advocated in Thorpe's Dictionary, viz., to allow the contents of the instrument to drain, and then touch the surface of the liquid once with the point. The most accurate pipettes, however, were probably those having two marks, one above and the other below the bulb.

Dr. Morgan said that the error in regard to weights was very much reduced if one operator followed his work right through with one set of weights. It was not desirable to have sets of weights by various makers in a laboratory.

Mr. Hehner said that he had never found a serious error in the calibration of pipettes. Standard solutions were mostly exceedingly dilute, compared with the strong sulphuric acid to which Mr. Richmond referred. No doubt for very accurate work, where it was necessary to reduce weights to vacuum value, it was advisable to check everything, but, for ordinary work, errors due to weights which had become a little worn were more or less compensated by other errors. He did not wish to be understood to say that, in his opinion, it was desirable to use very highly-worn weights, but it must be within the experience of most chemists that good weights which had become black and tarnished, when tested were found to be still accurate.

Mr. Richmond said that he wished to draw attention to the importance of accuracy in detail; an analyst who took the small precautions of standardizing weights, etc., would undoubtedly furnish to his clients more reliable results than if he left such things to chance.

The following papers were also read: "The Analysis of Waters for Minute Quantities of Poisonous Metals," by E. Russell Budden; "Note on the Analysis of Phosphor Tin," by Frank L. Teed, Ph.D.; "Note on Lemon and Orange Peel," by E. G. Clayton. The publication of these is unavoidably held over.

NOTES FROM THE LABORATORY OF THE ROYAL AGRICULTURAL SOCIETY OF ENGLAND.

BY J. AUGUSTUS VOELCKER, PH.D., B.Sc.

(Read at the Meeting March 7th, 1894.)

I. THE OCCURRENCE OF A POISONOUS LEGUMINOUS SEED IN INDIAN PEAS.

"INDIAN peas" is a vague term applied almost indiscriminately to a number of different leguminous seeds imported from India, and used for feeding purposes. The principal pulses thus used are the following: Gram (*Cicer arietinum*); arhar, or *dāl* (*Cajanus indicus*); the common pea (*Pisum sativum*); and the field pea (*Pisum arvense*), the last two being generally known as *mattar*.

Previous to my visit to India in 1890, I had not unfrequently come across instances where injury to stock, and even death, had been attributed to the use of

some mixed feeding-cake or other ; but, on examining the cake, I had been unable to find anything to justify the suspicion. It is well known to agricultural chemists that one of the dangers of mixed feeding-cakes consists in their being made of materials originally unsound ; but after the constituents have been cooked and pressed together into cake, it has been impossible to know what they were like at first. Of late years, too, agricultural chemists have been careful to search for the possible presence, in mixed cakes, of seeds which are known to have poisonous properties. I might refer here to a paper read before this society by my late assistant, Dr. Leather, on the detection of castor-oil bean in feeding-cakes.

In several instances brought to my notice before I went to India, I found that farmers had been tempted by the low price of "Indian peas" to purchase these instead of home-grown peas, and had experienced considerable trouble with the stock to which these peas had been given ; in most cases symptoms of paralysis were shown. I was unable at the time to account for this, but so certain was I that the complaints had some real basis that I was led to regard "Indian peas" with considerable suspicion. Accordingly, when I went to India in 1890, I made a special point of inquiring into this matter ; and I believe that I have arrived at the right solution. Since my return in 1891 I have watched the subject further, and I have within quite recent times had three cases brought to my notice in which harm has been attributed to the use of mixed feeding-cakes or meals, my subsequent examination showing there to be present in them the seed of a vetchling called *Lathyrus sativus*. This was the same seed that I met with in India, and which is known there to possess poisonous qualities. A specimen of the seed is here shown. It has a very unusual and characteristic shape, being ovate, or wedge-shaped, flattened on two sides, and having a very mottled or "marbled" appearance, and also a continuous thin black line running over about two-thirds of the circumference of the seed. The colour is variable, generally grayish, but it ranges from yellowish-brown to nearly black, and all seeds show the marbling to a more or less extent. Its size is about that of our English pea, but the shape is very different, and the seed is not rounded at all.

Lathyrus sativus, Linn., or *khesdri* pulse, is a crop grown principally in Northern India, and mainly as a fodder crop. The plant is a procumbent or climbing herb, the crop looking like a short one of vetches. It is grown on poor land that will raise no other kind of pulse, and generally after much rain has fallen. Duthie and Fuller, in their book "Field and Garden Crops of the North-West Provinces," speak of it as a coarse kind of pea, notorious for its effect in producing paralysis if eaten in excess ; and they mention that the widespread occurrence of paralysis in Sindh, after a season of extensive inundations, in which *khesdri* was grown on an exceptionally large scale, was due to the consumption of it, and also that cases of paralysis are far from uncommon in villages where *khesdri* forms an important item of diet. A case is cited where, at the military station of Almora, some cases of paralysis were traced to the fraudulent admixture of *khesdri* with the gram supplied for the use of the troops.

Colonel Sleeman also writes upon the ill effects of a large consumption of the grain in the province of Oudh in the years 1831 and 1832. Many of the people lost the use of their lower limbs entirely, and, when once attacked, never recovered completely. He adds that "it is the prevailing opinion of the natives throughout

the country that both horses and bullocks which have been much fed upon *khesdri* are liable to lose the use of their limbs; but if the poisonous qualities abound more in the grain than in the stalk or the leaves, man, who eats nothing but the grain, must be more liable to suffer from the use of this food than beasts which eat it merely as they eat grass or hay." The fact that the crop is principally used as a fodder crop is the probable explanation of the poisonous properties of the grain, when used as food for cattle, not being so well known among us.

In Dr. George Watt's "Dictionary of the Economic Products of India," it is pointed out that "an analysis by Astier has revealed the presence in the grain of a volatile liquid alkaloid, probably produced by some proteid ferment, which exhibits the toxic effects of the seeds, and the action of which is destroyed by heat." Numerous cases quoted in Dr. Watt's "Dictionary" leave no doubt as to the poisonous effects produced by the too free use of the grain. In horses, paralysis of the hinder extremities takes place, as also an affection of the larynx, ending in asphyxia and death. To the particular condition produced by this over-feeding, the name "lathyrismus" has been given. Dr. Watt refers to the importance of the fact that the alkaloid recently isolated is volatile, and that while in grain cooked at a high temperature it may be driven off, yet when the seed is cooked at a low temperature, or when used as grain for cattle, it may retain its poisonous properties. In this way the somewhat uncertain and variable effects of the grain may be explained.

I saw the crop growing in India myself, and made inquiries about it on the spot. These were quite in confirmation of the remarks made above, and I obtained also there the specimens of the seed which I now produce.

In the first of the three cases lately brought to my notice, in which poisonous results were observed from the use of this seed, the farmer complained that he had lost a quantity of beasts and sheep that had been eating a certain mixed feeding-cake. In the cake I found present seeds of the vetchling *Lathyrus sativus*; but, inasmuch as it transpired in the course of the inquiry that some of the cattle had been eating acorns, it was impossible to attribute the loss directly to the presence of the vetchling, and to say whether death resulted from it or from acorn poisoning. In a second instance I was informed by a firm of cake-makers, whom I had previously warned against buying "Indian peas," that they had inadvertently bought some, and had used them in the manufacture of a small lot of mixed cake. Within two days of sending out the delivery they had a complaint about it, and at once recalled the cake when they found the kind of peas that had been used in the manufacture. On examining the peas used, I found the *Lathyrus sativus* present.

Not above a fortnight ago I received a letter from a veterinary surgeon, stating that a farmer in his neighbourhood had, previous to his visit, lost two milch cows, and that on his arrival he had found four other cows unwell. "The most conspicuous symptoms," he added, "were paralysis." The cows had been feeding on a mixed meal got from a dealer in the neighbourhood, and a sample was sent up to me for analysis. I have here some of it, and a short examination showed me that there were present in it seeds of the *Lathyrus sativus*.

I was not able to ascertain more as to the origin of the meal than that it contained a portion of pea-meal, but I could not hear where the peas came from.

Lastly, I have at the present time a sample of mixed feeding-cake to examine, in which I have noticed whole seeds of the *Lathyrus sativus* present.

I have little doubt that in several cases where harm has been attributed to the use of mixed feeding-cakes or meals, or to that of Indian peas, the cause might have been found in the presence of this poisonous seed *Lathyrus sativus*. It is not distinguishable from other pulses by any peculiar microscopical appearances, or by any difference of its starch; but the marbling is very characteristic, as is also the shape of the seed, and the thin black line running nearly round the seed. It is well to point out that seeds of a greenish colour, and having a similar marbling to *Lathyrus sativus*, frequently occur among the ordinary varieties of pea, *Pisum sativum*, or *Pisum arvense*, and must not be confounded with it. The *Lathyrus* seed is flattened in shape, and not rounded like the seeds of *Pisum sativum* and *Pisum arvense*.

DISCUSSION.

The Chairman (Mr. A. H. Allen) said that Dr. Voelcker had undoubtedly brought forward a very important subject. Analysts ought to be very much indebted to anyone who would point out these minute differences between innocent seeds and poisonous species.

Dr. Bernard Dyer said that Dr. Voelcker was the first to call his attention to this matter. He had certainly met with several cases in which very virulent poisoning had followed the eating of peas coming from the East. He remembered a case of Persian peas coming into the market. A miller ground them into meal, which he sold to several customers, and with which he commenced feeding his own pigs. His pigs died promptly, and he telegraphed to his various customers, but in several cases their pigs were also dead or dying. Not long ago he had had brought to him some water by a firm of wharfingers to see if anything poisonous had been put into it, because their horses had died and others had been unwell, and it was thought that the cisterns had been poisoned. There was nothing abnormal in the water. He found, however, they had been feeding the horses on Indian peas. Unfortunately they had none of the peas left, so that it was impossible to find out whether any of them were poisonous; but the dates between which they were given to the horses coincided, he understood, with the time during which the illness and death of the horses took place.

Dr. Dupré said that the fact that the boiling sometimes rendered the peas innocuous, did not, in his opinion, point to a volatile alkaloid being present, as one could scarcely imagine that a short heating would expel all the volatile alkali. The effects observed rather indicated poisoning by organisms. In a case of poisoning which occurred some years ago, and which was due to the eating of bread-and-butter pudding, it was found that the puddings were made from the refuse bread of a coffee-house, some of which had become mouldy. One of the puddings which had been well baked proved harmless, the second was imperfectly cooked, and was poisonous. That the poisonous character of these peas might be due to the production of poisons by organisms, seemed to him worthy of consideration.

Mr. M. A. Adams thought it was a remarkable thing that of an alkaloid it should

have the property of producing paralysis. It had struck him that the poisonous substance must be rather of the nature of an albumose than an alkaloid. The fact of the seed containing a paralytic poison suggested to his mind that it came within the class of albumose substances. Until something was done, chemically, to prove the contrary, he was disposed to the idea that probably the poison was foreign to the seed.

Mr. John Hughes asked Dr. Voelcker if he had made any estimation of the amount of the poisonous seeds present in the feeding cake.

Dr. Frank L. Teed believed that Dr. Voelcker had commenced by saying that these poisonous seeds were grown as a fodder crop. The matter therefore seemed to him to require some sort of explanation, because it seemed very strange that a crop which was known to be poisonous should be grown. He (Dr. Teed) inferred that all that Dr. Voelcker had in any way demonstrated was that Indian peas were sometimes dangerous. He thought the explanation suggested by Dr. Dupré would have great weight, and that explanation ought to be thoroughly investigated before the seed of a non-poisonous plant received the condemnation which Dr. Voelcker wished to place on it from apparently insufficient evidence. He wished to know if Dr. Voelcker had any direct evidence of the poisonous action of these seeds.

Dr. Dyer anticipated Dr. Voelcker's reply by pointing out that, in our own country, and more especially in Ireland, a fodder crop very largely used for horses was grown, viz., furze or gorse. Although this was a very favourite fodder for horses, the seed, nevertheless, was virulently poisonous, containing the alkaloid ulexine, similar in its toxicological action to the laburnum poison.

Mr. Otto Hehner wished to ask Dr. Voelcker one question, from an analytical point of view. Dr. Voelcker had informed the Society that there was no microscopic difference in the structure of these seeds, and he seemed to recognise them solely by the form, the shape, and the wrinkling or the corrugations. If these seeds were crushed or made into meal, which was a very probable contingency, how would the analyst be able to deal with the matter?

Mr. H. Droop Richmond drew attention to the fact that Dr. Vaughan some time ago investigated some cases of ice-cream poisoning in the United States. It was found that a poisonous alkaloid had been developed in the ice cream; this was isolated by Dr. Vaughan, and named "tyrotoxinon." The identity of tyrotoxinon with diazobenzene butyrate had since been established.

Mr. F. H. Perry Coste said that, in connection with Mr. Richmond's view, danger from poison could be got rid of by roasting in the case of acorns; which had been, he believed, cooked and eaten by various barbarous nations, who presumably would not eat them if they were poisonous. He did not see that there was any *a priori* improbability of an alkaloid being present in this case. Besides, as Dr. Dyer had mentioned, an alkaloid had been isolated from *Ulex*. It was also known that the seeds of the Laburnum were exceedingly poisonous, and had sometimes been eaten by children with fatal results.

Dr. Voelcker quite agreed that only imperfect knowledge of the subject at

present existed. It was probable that the poison was either an alkaloid or an albuminoid ferment. He had not estimated the actual quantities of the bean present in different foods; the difficulty was to judge from the amount of the husk how much of the original seed had been present. In regard to what Dr. Teed had said, from an agricultural point of view no explanation was needed. He would cite the case of a mustard crop, which was injurious as regards the seed, but quite harmless when used as a green crop. Cases of paralysis were frequent in the districts where the *Lathyrus* was cultivated. It was essentially a matter of the quantity present; a small quantity might not produce paralysis.

II. ANALYSES OF WATERS FROM WELLS IN CLOSE PROXIMITY TO CHURCHYARDS.

It is not always that one can obtain definite information regarding the surroundings of wells, the water from which one is called upon to analyze, and when the particulars are forthcoming the chief points brought out by the analyses may be found useful for future guidance. Having recently analyzed three waters which I reported on adversely, and then finding that the wells were in close proximity to churchyards, I thought it might not be devoid of interest if I gave the analyses. The results were as follows:

	I.	II.	III.
	Grains per Gall.	Grains per Gall.	Grains per Gall.
Total solids...	23.52	38.36	69.99
Oxygen absorbed039	.066	.055
Chlorine ...	2.28	4.02	6.78
equal to Sodium chloride...	3.65	6.57	11.07
Nitric Acid ...	3.67	5.13	12.87
Free Ammonia006	.0005	.0005
Albuminoid Ammonia006	.013	.013
Phosphoric Acid29	.34	.52
equal to Phosphate of Lime	.59	.74	1.13

The wells from which waters I. and II. came were from the same place, and only about 80 yards apart. Water No. I. was not clear, was somewhat yellow-coloured, and had some amount of suspended matter. It left on evaporation to dryness a yellow residue. As the analysis showed, it was a water clearly open to grave suspicion. Water No. II. was free from any suspended matter or deposit, but was of a bright yellow colour. The chlorides and nitrates, as well as the total solids, were considerably more than in No. I., and the water was clearly badly polluted. On inquiry I found that both wells were within 25 to 30 feet of a churchyard, and that in a family which had drunk of the No. II. water, one, and it was believed two, members had died within the past three weeks of pronounced diphtheria. On inquiring further, so as to ascertain, if possible, the cause of the differences shown in the two analyses, I was told that while well No. II. was only 50 feet distant from several recent burials, well No. I. was 140 feet from one recent burial. The subsoil was hard gravel.

In the case of water No. III. the results were even more alarming. The water was of a yellowish colour, but was quite clear, and free from deposit. I was informed that the well was 23 feet deep, there being 8 feet of water in it; that a churchyard was only 100 feet distant, and a closet-drain 80 feet off. There had been illness here also from drinking the water. This water contained a considerable quantity of magnesia. Besides the high results in chlorides and nitrates shown in these analyses, it is be noticeable that in two out of the three cases a mere trace only of free ammonia was shown; and these instances prove that the significance of nitrates, even when a minute quantity only of ammonia is present, is very great, and ought never to be overlooked. I thought it desirable also to determine the phosphoric acid in the samples, and the results given are very high, as one might expect in the case of polluted water-supplies.

DISCUSSION.

Mr. Alfred H. Allen remarked that those analysts who were in the habit of making daily examinations of water for sanitary purposes were perfectly aware of the presence of nitrates in impure waters of this kind, although, unfortunately, their presence and indications were ignored by certain much-quoted authorities, with very great harm to the country generally. It was not at all unusual in his experience to find waters almost destitute of free ammonia, and yet containing large amounts of nitrates and chlorides. As to the phosphoric acid, he found that it was present very frequently in traces. He had, however, ceased to rely on it as an indication of contamination of the water, but he had never found anything like the amount stated by Dr. Voelcker. No doubt the proximity of the churchyard might account for these amounts.

Mr. M. A. Adams wished to ask two questions—first, as to the physical relation between the wells and the churchyard and the kind of well; and, secondly, as to the nature of the disease which was attributed to the drinking of the waters. He might say that from his experience of cases of diphtheria, he considered that this was not a water-borne disease. He would like Dr. Voelcker to tell him whether there had been any other disease in any way traceable to the waters. If so, it was important to know how the association between the waters and the disease was traced.

Dr. Teed asked Dr. Voelcker whether there was any other source of contamination in these waters; as from his experience one of them certainly appeared to be contaminated with sewage, and had not the characteristics of a cemetery water. Cemetery effluents were very rich in organic matter, but not in chlorine.

Mr. Benedict Kitto asked whether the waters were taken anywhere near the sea-coast, because he very frequently had such waters. Though they contained very high amounts of chlorides indeed, he passed them as quite safe for drinking purposes.

Mr. H. Droop Richmond asked Dr. Voelcker if he knew whether the fields in the vicinity had been treated with artificial fertilizers, because in one case he found a water containing a very large amount of phosphoric acid and nitrates, traceable to artificial fertilizers used on the fields, and the water was proved not to be contaminated except in this way.

Dr. Voelcker, in reply, said that on the medical side of the question he could say nothing. He had received the waters to examine, and he had reported on them. Subsequently, as a matter of interest, he had inquired whether there was anything to account for the state of things found. As regards the position of the wells, the first two were in Worcestershire, and the other at Tewkesbury, in Somersetshire. The soil in the first two cases was a sandy gravel, but he had no information as to the soil from which the last came. It was evidently a very much harder water, and there was a good deal of sulphate of lime in it, such as might be found in Somersetshire. In reply to Mr. Richmond, he did not know how a distinction could be drawn between the bones that lay in a churchyard and bones that might have been used as manure on a field. They both came under the category of manure, so that their effects might be alike, whether proceeding from the field or from the churchyard.

The Analysis of Rubber Goods. C. A. Lobry de Bruyn and F. H. van Leeut. (*Chem. Zeit.*, 1894, xviii., 309-312.)—The authors review various methods hitherto in vogue and dismiss many as illusory, notably such as assume that a rubber of low specific gravity, or one containing but little mineral matter, is necessarily of good quality—an invalid assumption in view of the fact that rubber containing surrogate may answer tests of this kind. An empirical method adopted by the Admiralty and capable of yielding useful data, consists in heating the rubber goods to be tested, dry to 135° C., and, with water in a sealed tube, to 170° C. The test discriminates between pure rubber of good quality, and that which is genuine but of poor quality, from the presence in it of an undue proportion of rubber resin, and also that which is adulterated with surrogate. Vulcanized Para rubber, alone and mixed with mineral ingredients (*e.g.*, zinc oxide), withstands exposure to the temperatures quoted for 2-4 hours without perceptible deterioration. Inferior goods are more or less damaged by the treatment, becoming hard and brittle and losing their elasticity, or swelling and fusing. The dry-heating test is carried out so that the loss of weight of the rubber is determined at the same time. Good vulcanized rubber, cut into thin shreds, loses 2 per cent. of its weight as a maximum at 135° C., the loss usually being less than 1.5 per cent. Thus a specimen of pure Para rubber lost 0.15 per cent. after one hour, and 0.25 per cent. after three hours; a sample from Mozambique 0.2 and 1.3 per cent. at the same periods, while the corresponding figures for a Borneo rubber were 1.7 and 3 per cent. The percentages of rubber resin in these three samples were 5, 6.5 and 36 per cent. respectively.

In judging rubber from its loss of weight it must be remembered that many common rubbers containing surrogate give low figures like genuine rubber containing a moderate proportion of rubber resin.

In applying Henriques' method for the detection of saponifiable constituents in rubber by extraction with alcoholic alkali (*THE ANALYST*, xviii. 13), the existence of resinous substances naturally present in rubber and soluble in the same menstruum must not be forgotten. As much as 40 per cent. of the total rubber substance may consist of bodies of this class, and their nature varies to some extent with the origin of the rubber of which they form part. The authors, while acknowledging the value

of Henriques' work, state that in their opinion the methods laid down by him are too lengthy for use when a rapid decision concerning the quality of a number of samples of rubber has to be arrived at, and append the results of some simple tests of a number of rubber articles of different sorts (*v. i.*). They note that when detailing the composition of rubber which is to be manufactured according to a definite specification, zinc oxide may be conveniently adopted as the mineral constituent, unless the rubber is to come into contact with acid liquids, when barium sulphate is a more suitable "filling" material. Zinc oxide is especially objectionable in laboratory corks that are used in such processes as the determination of an acetyl group in an ester by digestion for some six hours with $\frac{N}{10}$ sulphuric acid, low results being obtained. Chalk, which was present in a sample of rubber tubing to the extent of 70 per cent., is equally obnoxious. In carrying out the tests on the samples quoted below, the ash was determined by gentle ignition without special oxidation and carbonation—practices sometimes resorted to—and results of fair constancy, sufficient for judging the quality of the goods, were obtained. The determination of the percentage of matter soluble in alcoholic soda was carried out in the manner prescribed by Henriques, and in the case of rubber containing mineral constituents was calculated on the ash-free material. The estimation of the loss on heating to 135° C. for two hours was usually performed only on rubber free from ash, and was made on pieces of rubber not more than 1 mm. thick, placed in a drying-oven previously heated to the required temperature. The test, consisting in heating the rubber with water under pressure, was carried out by placing the samples (not subdivided) in a strong copper or iron tube closed with a screw and capable of withstanding a pressure of at least ten atmospheres, about half or two-thirds full of water, raising the tube and its contents to 170° C., and maintaining this temperature for four hours.

In the selected list given below the samples are arranged in the order of their content of ash; those for which no ash is recorded were practically ash-free, *i.e.*, contained not more than 1 per cent. The specimens marked with an asterisk withstood both heat tests:

No.	Description.	Ash Per Cent.	Percentage soluble in 6 per cent. Alcoholic Soda, cal- culated on the Rubber Free from Ash.
1.*	... Vulcanized Para rubber, 6.4% S.	...	5
2.*	... Vulcanized Mozambique rubber, 5.5% S.	...	6.5
3.	... Vulcanized Borneo rubber, 8.3% S.	...	36
9.	... Old laboratory cork	... 4.5	7
10.*	... Tubing ("patent" rubber)	...	4.9
15.*	... Black rubber ring	... 6.5	0.7
17.	... Rubber disc	... 15.5	9
18.	... Rubber ring	... 15.5	23.3
23.	... Red tubing	... 24.5	12
25.	... Rubber mat	... 44	50
26.*	... Rubber cork (French make)	... 44.5	11.9
27.	... Rubber packing	... 45	23
36.*	... Rubber valve	... 51.5	4
60.	... Rubber late	... 71	21.2

Certain of these samples were examined for their loss on heating to 135° C. :

No.	Loss after One Hour.	Two Hours.	Three Hours.
1. ...	0·01% ...	— % ...	0·25%
2. ...	0·02 ...	— ...	1·3
3. ...	1·7 ...	— ...	3
15. ...	1·1 ...	2·5 ...	—
18. ...	— ...	3 ...	—
25. ...	— ...	— ...	4

It is evident from the foregoing figures that a genuine rubber containing little rubber resin but much ash resists destructive influences better than an adulterated or resinous sample (yielding a large quantity of extractive matter on digestion with caustic soda), even though the latter be fairly free from ash.

White and brown surrogates lose 2 to 2·5 per cent. on heating to 135° C., but do not fuse at this temperature, and indeed are but little altered ; when subjected to the hot-water test, however, they fuse, darken, and decompose with the production of evil-smelling sulphur compounds. According to the authors' experience, rubber adulterated with surrogate does not stand the hot-water test. As stated above, it is generally true that samples yielding only small quantities of extract on treatment with alcoholic soda withstand the heat tests, but the rule is not universal. The age of the rubber is a factor that cannot be overlooked, as it is well known that even good rubber after years of use becomes much deteriorated. The authors call attention to the fact that the manufacture of rubber goods specially adapted for different laboratory purposes is very desirable. Thus corks and tubing containing much insoluble mineral matter (*e.g.*, BaSO₄) would be useful for purposes where contact with corrosive liquids and gases is inevitable, while all other laboratory rubber articles should be of pure ash-free rubber.

B. B.

The Analysis of Rubber Goods. R. Henriques. (*Chem. Zeit.*, 1894, xviii., 411, 412, and 442-444.)—The author has continued his endeavours to arrive at reliable general methods for the analysis of rubber goods ; these methods have been already abstracted into the ANALYST. In his former work he elaborated processes for the separation and estimation of fatty oils and oil surrogates in rubber, and now he has been able to check the accuracy of these methods by the analysis of rubber goods of known composition. Three samples—one containing brown surrogate, another white surrogate, and the third rape-oil—were prepared by a friendly manufacturer and analyzed by the author, with the results given below.

Before detailing the result of this work, it may be remarked that a minor improvement in the method originally used has been made. The fundamental separation of surrogate from rubber depends on the saponification of the former, and it is found that the rubber obstinately retains a certain amount of alkali, necessitating a correction in the estimation of the true rubber substance by determining both the total increase of ash and the sulphur contained therein. This complication can be in great measure avoided by boiling out the rubber, after treatment with alcoholic alkali to remove surrogate, with dilute hydrochloric acid. Not quite all the alkali is thus removed, some 2 to 3 per cent. remaining ; but the quantity of sulphur retained

is negligible, and the difference between the original ash and that obtained after the successive treatments with alkali and acid, added to the apparent value for the soluble surrogate, gives the true amount thus extracted. This modification, involving the use of an acid solvent, is applicable in the case of rubber containing soluble inorganic constituents, as these are first removed by treatment with acid before the determination of the surrogate is begun. The following data are required in the determination of the composition of rubber goods containing surrogate (or oil) rubber and sulphur: (*a*) Total sulphur, (*b*) total ash, (*c*) residue after extraction with alkali, (*d*) sulphur in this residue (calculated on the original rubber), (*e*) ash in this residue (calculated on the original rubber), (*f*) sulphur in the extracted fatty acids. For the determination of *c*, 1.5 to 2 grammes of the substance should be boiled for 2 to 3 hours with alcoholic soda, the extract being poured off and the treatment repeated. A mean correction of 2.5 per cent. is made for the amount of true rubber dissolved, this value being the average of ten determinations with various genuine samples of rubber, which gave figures ranging from 0.9 to 3.5 per cent. The determination of the other data (*a*, *b*, etc.) is performed according to the prescription already detailed by the author in his former papers. Then, calling the percentage of pure rubber *x*, and that of the fatty acids *y*, we have:

$$\frac{2.5x}{100} + y = 100 - c + (c - b) - (a - d)$$

and

$$x + y = 100 - (a + b)$$

solving which we obtain:

$$x = \frac{100}{97.5} (c - d - e)$$

$$y = 100 - (a + b + x).$$

The value of *f* is obtained from the facts, arrived at in an earlier part of the investigation, that the sulphur content of white surrogate is identical with that of its fatty acids, and that the sulphur content of brown surrogate is on the average 1.5 per cent. higher than that of its fatty acids. Finally, the sulphur used for vulcanizing is obtained as the difference between the total sulphur and that contained in the surrogate. The analyses of the three samples mentioned above were conducted on these lines, the figures obtained being:

	I. Rubber + Brown Surrogate.	II. Rubber + White Surrogate.	III. Rubber + Oil.
(<i>a</i>)	19.46	11.38	21.55
(<i>b</i>)	0.72	0.40	0.40
(<i>c</i>)	68.37	67.68	68.40
(<i>d</i>)	2.64	1.51	3.06
(<i>e</i>)	2.70	2.81	3.17
(<i>f</i>)	16.60	9.27	3.20
Chlorine.	absent.	much present.	absent.

The composition of the three samples deduced from these figures and arrived at synthetically are compared below:

I. *Rubber + Brown Surrogate.*

Analysis.				Synthesis.
Sulphur	16.13 per cent.	16.4 per cent.
Brown surrogate	18.50 "	19.3 "
Rubber	64.65 "	64.3 "
Ash	0.73 "	—

II. *Rubber + White Surrogate.*

Analysis.				Synthesis.
Sulphur	9.19 per cent.	9.6 per cent.
White surrogate	25.43 "	25.8 "
Rubber	64.98 "	64.6 "
Ash	0.40 "	—

III. *Rubber + Oil.*

Analysis.				Synthesis.
Sulphur	21.55 per cent.	23.5 per cent.
Oil	20.42 "	17.7 "
Rubber	57.63 "	58.8 "
Ash	0.40 "	—

It is evident from the foregoing figures that the concordance between the calculated and ascertained results is good in the case of Samples I. and II. containing white and brown surrogate, but that it is less close for the mixture of rubber and oil (No. III.). The author attributes this divergence less to error of analyses than to the character of the sample analysed. Although of normal aspect, it was thoroughly porous, as if an evolution of gas had occurred during vulcanization (causing loss of sulphur as H_2S). It is also an acknowledged fact that fatty oils strongly attack rubber at high temperatures, and resolve it into products soluble in alkali. This accounts for the presence of sulphur in the fatty acids in not inconsiderable amount (3.20 per cent.), although the oil originally used was necessarily free from sulphur. The discrepancies thus occasioned are not of much practical importance, as rubber containing considerable quantities of fatty oil is not met with in commerce, the only sample which the author has examined being an unvulcanized mixture for the preparation of hard rubber, the oil in which would be converted into surrogate in the act of vulcanization by the excess of sulphur present.

A contribution to existing methods for separating rubber from fatty oils has been made by Holde (*THE ANALYST*, xix., 43), who uses ether-alcohol as a solvent for the oil. The author has confirmed Holde's statement that the average amount of matter dissolved from pure rubber by ether-alcohol is 5 per cent., but suggests a slight modification of procedure. Instead of washing the comminuted rubber on the filter with the solvent, the material under examination should be digested with half the total quantity of ether-alcohol (sixty times the amount of rubber taken) in the cold for one hour, filtered, and washed with the remainder of the solvent. The weighing of the residual rubber is also stated to be simpler and quicker than the recovery of the oil. Should surrogate as well as oil be present, the method is not applicable, as the solubility of commercial surrogates in ether-alcohol varies widely. Thus white surrogate gave up 6.96 and 19.65 per cent., brown surrogate 11.05 and 6.69 per cent., castor-oil surrogate 9.85 per cent., and surrogate prepared according to German

patent 73045, 33.96 per cent. to ether-alcohol. It is of course possible that the substance thus extracted from these surrogates may be unaltered oil or its oxidation products which have escaped the action of the sulphur or sulphur chloride used in preparing the surrogate, but the practical result is the same, and discrimination between surrogate (with or without unacted-on oil) and oil added as such is rendered impracticable. On these grounds the author prefers his own methods. A better separation of oil and rubber might be effected by the use of the solvent proposed by Weber (*Zeit. f. angew. Chem.*, 1893, 631), consisting of two volumes of methyl alcohol and one volume of benzene; but the statement that this mixture dissolves no rubber must be accepted with reservation, the author having found it remove 4.9 per cent. (consisting half of rubber and half of sulphur) from a sample of pure vulcanized Para rubber. No organic solvent with which experiment has been made has been found to be quite without action on rubber.

Estimation of Asphalt and Lampblack in Rubber Goods.—The author alluded to this analytical problem in the earlier part of his investigations, but deferred its solution until the question of determining the commoner admixtures (*e.g.*, surrogate) had been settled. The following are the results at which he has now arrived.

Seeing that both asphalt and rubber are neutral substances remarkably resistant to most chemical agents, separation by means of a suitable solvent is the most promising method of dealing with them. The earlier experiments made in this direction were concerned with the separation of asphalt from unvulcanized rubber. Such organic solvents as chloroform and oil of turpentine, which dissolve asphalt without residue, and convert unvulcanized rubber into a gummy mass difficult to deal with, are therefore unsuitable. Nitro-benzene was found to be a good solvent for asphalt while attacking rubber in minor degree. One gramme of the finely-divided sample is digested in the cold with 30 c.c. of nitro-benzene for one hour, and the whole mass transferred to a filter, where the liquid is expressed as far as possible by means of a small pestle, and the residual rubber washed with a further 30 c.c. of the solvent. The rubber is washed off the filter by a jet of water into a porcelain dish, and the adhering nitro-benzene evaporated, its volatilization being aided by the concomitant water vapour. When all smell of nitro-benzene has ceased, the rubber is again filtered from the unevaporated portion of the water, dried and weighed. Should the rubber contain inorganic constituents which are soluble in water, the last filtration should be avoided, and the residual water evaporated to dryness.

Four samples of rubber were treated in this manner in order to ascertain what loss they suffered. Samples A and B, which were pure rolled raw rubber, lost 1.14 and 2.03 per cent. respectively, while C and D, which were pure "patent" plate rubber, lost 1.10 and 1.54 per cent. A mean correction of 1.5 per cent. must, therefore, be applied in estimating asphalt by this process. The validity of the method was verified by incorporating raw rubber with known quantities of asphalt dissolved in chloroform, drying the whole mass, and extracting it by means of nitro-benzene. The agreement between the found and calculated results was good.

The matter becomes slightly less simple in the case of vulcanized rubber, because of the presence of sulphur therein. The difficulty is overcome by removing the sulphur before determining the asphalt, this object being attained in the course of

the usual treatment with alcoholic alkali to extract surrogate. Provided the alcohol be evaporated before filtering after the alkali extraction, no asphalt is retained in solution. The constant for the loss of vulcanized rubber when treated with nitrobenzene is somewhat higher than that for raw rubber (1.5 per cent.), viz., 3 per cent. Using these precautions and this correction, a good agreement between the calculated and found content of asphalt in vulcanized rubber was obtained by the author. From hard rubber rather less matter is extracted than from soft rubber, but the difference is not great enough to invalidate the correction (3 per cent.) given above. The process is applicable to artificial asphalt made from coal-tar pitch, as well as to natural mineral asphalt. It must be understood that the method needs modification in the presence of mineral oils, paraffin, and similar unsaponifiable substances, which would be unattacked by the alcoholic alkali used to remove fatty oils and surrogate; but cases in which these bodies occur along with asphalt are rare, and can be dealt with as they arise.

Passing to the detection of lampblack in rubber goods, the following considerations serve for the foundation of a method. Experiments by the author confirm the formula $(C_{10}H_{16})_n$ as generally representative of the composition of pure rubber, the limits found being $C : H = 10 : 15.31$ and $C : H = 10 : 17.02$. Taking the mean ratio $C : H = 10 : 16$ as correct, the excess of carbon found by combustion over and above that necessary to provide for the hydrogen represents free carbon, and serves as a measure of the lampblack content. The cardinal failing of this method is that the hydrogen is the basis of calculation, and errors in its determination are largely multiplied in calculating therefrom the carbon present as rubber. The importance of this source of error is minimized in practice by the circumstance that rubber goods containing lampblack are almost certain to contain other adulterants, and an error in the estimation of the lampblack in the residual rubber after removal of surrogate, etc., becomes relatively less considerable when calculated on the original rubber article. (Bearing this in mind, it is obvious that an improvement in closeness of result can be effected by removing the bulk of the rubber before combustion by solution in mineral oil, as suggested by the author in his previous papers, always provided that the fraction of the rubber left undissolved preserves the ratio $C : H = 10 : 16$.) The author quotes several analyses which prove the substantial accuracy of the process. He concludes the paper by an example of the analysis of a complex specimen of commercial rubber, viz., an india-rubber overshoe. Before describing the actual scheme of analysis, he mentions that he now avoids the separation of the sulphur present as vulcanizing sulphur from that existing as sulphides and sulphates by means of solution of the rubber in petroleum, on the ground that the process is tedious and yields imperfect solution when the rubber is heavily weighted. It is preferable, therefore, to divide the rubber as finely as possible with a rasp, decompose the sulphides by treatment with acid, the H_2S evolved being determined, and estimate the soluble sulphates in the solution thus obtained. The residue can then contain sulphur only in the form of vulcanizing sulphur and insoluble sulphates (*e.g.*, $PbSO_4$ and $BaSO_4$), which are allowed for by calculation from their bases.

The rubber overshoe analyzed was of well-known make, and generally considered

to be of good quality. It was easily reduced to fine powder. One gramme was treated with HCl, and the total gases evolved (CO_2 and H_2S) weighed, the lead sulphide present being completely decomposed. The residue was repeatedly extracted with dilute acid, and the bases determined in the total extract, which was free from sulphate. The total sulphur in the residue was determined by the method previously described by the author, and a trace of lead, which had existed as sulphate (derived from the lead sulphide by accidental oxidation), obtained in the same portion, as well as a little silica and alumina. Another portion of the rubber (1 gramme) was used for the determination of the sulphur in all forms, the difference between this and that in the insoluble residue giving the sulphur evolved as H_2S , and therefore the CO_2 estimated at the same time. A third portion of 5 grammes was treated with acid, filtered, washed, and weighed. Surrogate was looked for in part of the residue, and only a trace found; the small quantity detected may have been nothing but oily matter naturally present in the rubber. Another part of the same residue was used for the determination of asphalt and lampblack in the manner described above. The following are the figures obtained :

Lead oxide	8.45	per cent.
Calcium carbonate	47.81	"
Ferric oxide and alumina...	1.70	"
Silica	0.44	"
Vulcanizing sulphur*	1.50	"
Oily matter	0.95	"
Asphalt	8.46	"
Lampblack	0.39	"
Caoutchouc	30.30	"
Total	100.00	

The small percentage of sulphur is noteworthy, as is also the fact that the greater part exists as lead sulphide. As litharge, and not lead sulphide, is used in the manufacture, the function of about one-half appears to be to unite this with sulphur—3.94 per cent. PbO being thus accounted for. The remaining sulphur is only about $2\frac{1}{2}$ per cent. of the caoutchouc, *i.e.*, the quantity absolutely requisite for vulcanization.

B. B.

Determination of Chromium in Chromium Steel. J. Spüller and S. Kalman. (*Chem. Zeit.*, 1893, xvii. 1360, 1361 and 1412.)—The authors have extended their method for the determination of chromium in ferrochromium (*ANALYST* xviii., 251) to the determination of the same element in chromium steel.

Two grammes of steel are dissolved in a round-bottomed porcelain basin in 16 c.c. of sulphuric acid (1:5 by volume), the solution evaporated to dryness and heated strongly, the crust of sulphates being broken up by means of a glass rod. The dried residue is transferred to a silver dish, intimately mixed with 4 grammes of sodium peroxide and 8 grammes of caustic soda, and the mass gently heated until the evolu-

* Of this 0.61 per cent. was present as PbS , 0.08 per cent. as PbSO_4 , and 0.81 per cent. as free sulphur and sulphur combined with caoutchouc.

tion of oxygen, which at first takes place from the action of the sulphates on the sodium peroxide, has ceased. The melt should be stirred with a silver spatula during this part of the process to prevent its frothing over. The temperature is then raised and the fusion continued for fifteen minutes, at the end of which time 4 grammes of sodium peroxide are added, followed by another portion of 2 grammes after another twenty minutes. The whole of the chromium is converted into sodium chromate in a time not exceeding one hour and a quarter for the whole series of operations. The silver dish with its contents is allowed to cool until it has reached a temperature of 40° to 50°C, when it is wiped and placed in a roomy porcelain basin and the melt exhausted with water. The sodium manganate and ferrate are removed, as in the case of ferrochromium, by treatment with small successive doses of sodium peroxide, and the excess of sodium peroxide decomposed by passing carbon dioxide through the liquid while hot. The solution of the melt, containing oxides of iron, manganese and aluminium in suspension, is made up to one litre, well-mixed, and an aliquot portion, *e.g.*, 250 c.c. to 500 c.c., according to the richness of the steel in chromium taken for analysis. The rest of the process for the volumetric determination of chromium is conducted as in the analysis of ferrochromium described in the abstract already quoted, save that 25—50 c.c. of sulphuric acid (1:5) are used instead of 20 c.c. The mass of sulphates which has to be transferred from the vessel in which the steel is dissolved to that in which the fusion is conducted, is generally easy to detach from the former and leaves but little sticking to the sides. Any traces that may adhere should be wiped out with a small piece of damp filter-paper, incinerated and added to the main quantity. Should the sulphates have been insufficiently dried, the free sulphuric acid accompanying them will decompose much sodium peroxide before the temperature of fusion is reached, so that the addition of a further quantity of the oxidant is necessary.

The method was tested with two samples of chromium steel which nominally contained 1.5 and 4 per cent of chromium. The results were satisfactory, being for the first sample 1.35 and 1.40 per cent., as against 1.43 determined by Blair's gravimetric method (which is usually accepted as accurate though tedious), and 4.08 and 4.10 per cent. instead of 4.10 per cent. by the same check process. The use of the volumetric process described by the authors for determining chromium is not interfered with by the presence of tungsten.

An attempt was made to estimate the amount of chromium in steel directly, without previous solution in acid. It was found that the usual mixture of sodium peroxide and caustic soda was ineffective, and that even sodium peroxide alone failed to attack the metal completely. By hardening the steel, by plunging it while red-hot into cold water, so that it could be broken with the hammer and afterwards reduced to powder in a steel mortar, better results were attained. The subdivision of the metal was best effected by heating the coarse powder obtained as already described, in a Rose's crucible in a stream of oxygen at a low temperature for a period of ten minutes, and rubbing down the oxidized metal in an agate mortar. By treating the product with a mixture of sodium peroxide and caustic soda in the proportion of 10 grammes of the former and 4 grammes of the latter to 2 grammes of the metal and adding another 2 grammes of peroxide during the fusion, complete attack of the

steel can be ensured. The time and labour consumed in comminution are, however, greater than would be required for the solution of the steel in acid.

The use of sodium peroxide is practicable for ferrosilicon and ferrotungsten, and possibly also for ferroaluminium and ferrotitanium. B. B.

The Methods of Testing Fats and Oils. E. Milliau. (*Jour. Franklin Inst.*, 1898, cxxxvi. 376-388 and 433-442.)—This paper contains a description of the methods adopted in the Government Testing Laboratory at Marseilles. The majority of the tests are well known, inasmuch as they differ but slightly from those described in Allen's "Commercial Organic Analysis," vol. ii. The following may be culled, as being of more recent origin :

Examination of Olive-oil for Earth-nut-oil. — Renard's method is modified as follows: 20 grammes of the oil are saponified by 20 c.c. of a caustic soda solution of 36° B., diluted in 100 c.c. alcohol 90 per cent. (*sic*). The soap is precipitated by a 50 per cent. alcoholic solution of neutral lead acetate. The liquid is decanted while warm, and the residue washed with alcohol, ground in a mortar, and agitated with 200 c.c. of ether. The grinding and agitation are repeated, in order to fully extract lead oleate. The residue is then put in a porcelain dish containing 2 or 3 litres of water and 50 c.c. of hydrochloric acid. When decomposition is complete, the solution is decanted, and the fatty acids washed with water, dried in an oven, and dissolved in 40 c.c. of absolute alcohol; a drop of hydrochloric acid is added, and the liquid chilled to 15°. Arachidic acid crystals separate if earth-nut-oil be present.* These are washed first with 90 per cent. alcohol, and then with 70 per cent. alcohol, two lots of 20 c.c. being used in the first case, and three lots of 20 c.c. in the second. The acids are warmed slightly and treated with boiling absolute alcohol. After filtration the alcohol is evaporated, and the residue heated at 100° until constant in weight; it must then melt at 73° to 75°, in order that it may be fully identified as arachidic acid.

In conducting the usual hydrochloric acid and sugar test for sesame-oil in olive-oil the free fatty acids must be used, because the colouring matter of the olive, always present in the oil, gives a reaction with hydrochloric acid and sugar which is indistinguishable from that of sesame-oil.

The author cannot admit Bechi's method for detecting cotton-seed-oil. Instead, he heats 15 c.c. of the oil to about 110°, and pours in, slowly, a mixture of 10 c.c. of a solution of caustic soda of 36° B. with 10 c.c. of alcohol. When the mass boils, 150 c.c. of hot distilled water are added, and the boiling is continued to expel alcohol. The fatty acids are liberated with 10 per cent. sulphuric acid, and immediately collected by a small platinum spoon. They are washed by shaking several times in a test-tube with water; the water is drained off, and the acids poured into a tube 9 cm. long by 2.5 cm. diameter; 15 c.c. of 95 per cent. alcohol and 2 c.c. of a 3 per cent. solution of silver nitrate are added—the tube protected from the light, and heated at 90°, until about one-third of the alcohol is expelled. This is replaced by 10 c.c. of distilled water, and the heating is continued for a few minutes. The black coloration of the fatty acids is now detected if cotton-seed-oil, in any proportion, was originally present.

* Fonzio has recently shown that rape-oil contains about 4 per cent. of arachidic acid.—A. G. B.

For the rapid identification of castor-oil in sesame-oil, 10 grammes are shaken with 4 drops of sulphuric acid (66° B.); a drop of nitric acid (40° B.) is added, and the mixture is shaken violently. Pure sesame-oil blackens immediately, whilst that containing castor-oil remains turbid yellow.

Coco-nut and palm-nut oils are entirely soluble in absolute alcohol; at 30° to 31° the former requires two volumes and the latter four volumes for complete solution. The smallest addition of vegetable or animal oil impairs this solubility. To apply these facts the oil must first be purified by agitating 20 c.c. with 40 c.c. of 95 per cent. alcohol. Oils, such as castor and rosin oil, will be thus detected, for they are soluble in alcohol of this strength; moreover, mowrah and karite oils give a milky turbidity to the alcoholic stratum. Five c.c. of the purified oil are placed in a graduated test-tube, and 10 c.c. of absolute alcohol are added. The temperature is raised to 31°, the tube shaken violently for half a minute, and then immersed in a water-bath kept at a temperature slightly above that of the tube. Pure coco-nut-oil dissolves completely, and the solution remains clear. Any added oil causes precipitation: palm-nut-oil will precipitate from this solution of coco-nut-oil if it amounts to 20 per cent.; otherwise, the mass remains turbid.

Palm-nut-oil is similarly verified, 20 c.c. of absolute alcohol being substituted for 10 c.c. Five c.c. of palm-nut-oil, containing 20 per cent. or more of coco-nut-oil, dissolves in 15 c.c. of absolute alcohol. In the same proportions pure palm-nut-oil does not dissolve, and the mixture remains turbid; a mixture of these two oils, which would appear pure by the iodine absorption and saponification equivalent, would thus be detected by this method. The purity of coco-nut and palm-nut cake may also be determined by first extracting the oil by means of a solvent, and then operating on it in the manner described.

With regard to butter, the author maintains that the determination of the fixed and volatile acids, and of the solubility in alcoholic toluene, will serve to detect admixtures above 10 per cent. Below this point he does not believe that any adulteration would be profitable.

A. G. B.

Milk, Skim-Milk, and Whey. By C. B. Cochran. (*Amer. Chem. Soc. Jour.*, 1893, xv., 347-351.)—This paper contains some laudatory remarks concerning Richmond's milk scale, which, in the author's opinion, should be authoritatively adopted in the United States, as a check in the analysis of normal milk; chemists who obtain results not in accord with the formula should feel it incumbent on them to investigate the cause of the disagreement.

A number of skim-milks have been analyzed, with a view to ascertaining the specific gravity of casein in solution and the amount by which the specific gravity of the milk is lowered when 1 per cent. of casein is removed. The average of the results shows that each percentage of casein removed lowers the specific gravity of the solution 2.72 "degrees," and that the density of the casein in solution is 1.376.

The figures given for these values, respectively, by Richmond are 2.57° and 1.346; by Dupré, 2.55° and 1.34; by Hehner, 2.36° and 1.3106.

From his analysis of wheys, the author concludes that, when the coagulation is uniformly performed, the specific gravity and total solids of whey vary within

narrow limits, the former varying from 6.5 to 6.9 per cent., and the latter averaging 1.027.

The following table illustrates how a knowledge of the composition and specific gravity of the whey may serve as an aid in determining adulteration by dilution :

No.	Specific Gravity.	Total Solids.	Solids-not-Fat.	Specific Gravity of Whey.	Total Solids of Whey.
1.	1.0307	14.32	8.78	1.0280	—
2.	1.0315	12.05	8.55	1.0270	—
3.	1.0273	10.90	7.50	1.0230	—
4.	1.0204	12.05	6.15	1.0213	—
5.	1.0318	14.68	9.49	1.0255	5.89
6.	1.0310	12.05	8.45	1.0280	6.59
7.	1.0312	12.45	8.55	1.0270	6.35
8.	1.0320	12.90	8.80	1.0275	6.47
9.	1.0329	13.22	9.02	1.0273	6.42
10.	1.0324	12.98	8.88	1.0283	6.66
11.	1.0329	12.40	8.90	1.0270	6.35
12.	1.0278	8.81	8.71	1.0234	5.50

No. 1. A partially creamed sample of milk.

No. 2. Poor milk.

No. 3. Watered milk.

No. 4. Watered cream.

No. 5. Whey separated after milk had stood three weeks.

No. 12. Watered skim-milk.

A. G. B.

REVIEW.

HANDBUCH DER STICKSTOFFHALTIGEN ORTHOCONDENSATIONS PRODUCTE (Handbook of Nitrogenous Ortho-Condensation Products). By O. KÜHLING. Berlin : Oppenheim. Price 14 marks.

The *raison d'être* of this work shows the marvellous development in Germany of a branch of chemical technology, the origin of which may be traced to the publication of August Kekulé's theoretical views in 1865 on the constitution of benzene, the parent substance of the so-called aromatic compounds.

The subject-matter of this book covers 628 pages. Nitrogenous ortho-condensation products include a great number of the numerous artificially-prepared dyes, and many compounds of therapeutic value.

After a short introduction, the author devotes seventy pages to a general discussion on the constitution, properties, and syntheses of nitrogenous ortho-condensation products. The special part, which follows this, and occupies the remainder of the book, contains for each chapter a theoretical portion and a tabulated list of compounds, showing their constitutions, modes of preparation, and bibliographical references. No mention is made, however, of methods for the detection and estimation of the compounds in question. The rule, adopted by Beilstein in his well-known "Handbuch der Organischen Chemie," of only mentioning compounds of which analytical data have been furnished is carried out as far as possible, but exception is taken in this respect to certain technically-important substances. The technological value of the work is undoubtedly enhanced by references to the patent literature. The author invites his readers to notify errata to him, so that a second edition of the work as free as possible from errors may be prepared.

A. R. L.

THE ANALYST.

JUNE, 1894.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on May 2nd at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Alfred H. Allen took the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were duly elected : As Members, Edward M. Chaplin, Thompson's Yard, Westgate, Wakefield ; B. Henry Gerrans, 47, Aubert Park, High-bury, N. ; William Marshall, 15, West Street, Rochdale ; and B. H. Mumby, M.D., Portsmouth.

Mr. Harvey then read the following paper :

A SIMPLE APPARATUS FOR THE EXTRACTION FOR ANALYSIS OF GASES DISSOLVED IN WATER.

By SIDNEY HARVEY.

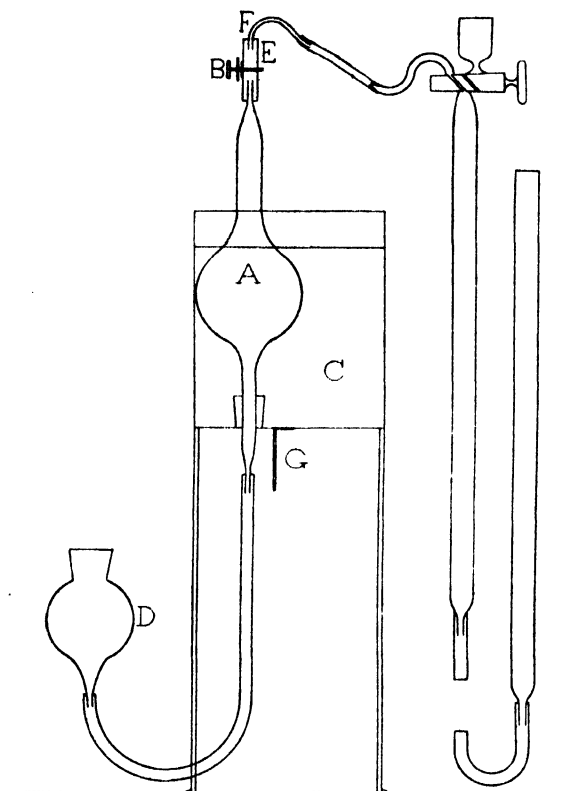
Having been engaged for several months past in an investigation in which the daily estimation of the gases dissolved in water was called for, specially as regards the amount of free CO_2 present, I deemed it desirable to confirm the results, obtained by the ordinary gravimetric and titration method, by the actual measurement of the gases themselves.

In doing this I was perplexed by the complicated character and unsatisfactory performance of various forms of apparatus proposed for the purpose.

Among the drawbacks attending the use of these may be mentioned, the uncertainty as to the exact amount of water operated upon ; the unavoidable escape of some of the water (when expanded) into some part of the apparatus where it fails to undergo efficient treatment ; the use of steam to expel air from the apparatus at the outset, and the consequent inconvenience due to the presence of the condensed water therefrom, either into the gas receiver or elsewhere, favouring re-absorption and consequently low results ; the number of junctions requiring careful securing against leakage.

After trying several forms of apparatus, I found one suggested by Thresh (*Journal Chemical Society*, Transactions, vol. xxxix., p. 399) to be the best for my purpose, but even in this ingenious arrangement I found it difficult to keep the perforated rubber stoppers secure throughout the operation. Profiting, however, by Dr. Thresh's suggestion, I ultimately constructed the present apparatus, which, while, no doubt, subject to criticism, has at least the merit of simplicity, and has proved very serviceable and manageable in my hands.

It consists of a globular, spindle-shaped glass vessel (A), having two opposite tapering necks, 13 to 14 inches long between extremities, the globular part $3\frac{1}{4}$ or $3\frac{1}{2}$ inches in diameter. The upper neck is somewhat enlarged, tapering gradually, the



lower is narrow and cylindrical; both end in a capillary bore with a swollen tip, in order that the rubber tubing may be securely affixed. The upper one has a short length of small-bore pressure tubing, securely tied and furnished with a screw clamp (B).

The apparatus is used as follows: In the first place the exact capacity of the vessel from end to end is ascertained once for all. It is then filled with the sample to be analyzed, the screw clamp (B) is closed, and the vessel carefully fixed upright in a tin water-bath (C) 6 inches diameter, 7 inches high, standing on legs 12 inches high, having an opening in the bottom closed by a perforated cork, and so arranged that while the lower stem of the spindle projects 2 inches below the bottom, the globular part is immersed in the bath itself.

The clamp is now opened, and about one-third of the water allowed to run out into a measured vessel. Of course, the amount deducted from the capacity of the globe when full gives the amount experimented upon. The lower end of the spindle has now two feet of small-bore pressure tubing slipped over it and secured. This tubing has a mercury reservoir (D) at the other end, and the latter suitably supported.

Clean pure mercury is now poured into the reservoir, the clamp (B) again opened, and the air, together with any bubbles, driven out, the water being allowed to follow to the upper end of the rubber tube (E). The clamp is now closed again. An ordinary nitrometer, having a bent capillary glass tube (F) affixed to the beak, is now filled with mercury, and the mercury forced to the end of the capillary tube, which is now thrust into the top of the rubber tubing and secured. The reservoir is now lowered, and the clamp cautiously opened in order to draw a little mercury sufficiently far to reach the lower end of the capillary tube. The clamp is now closed, the water-bath filled with cold water, and heat applied. To prevent the latter injuring the lower end of

the spindle, a metal curtain (a) is riveted on to the bottom of the bath so as to screen the glass from the flame, and for the same reason the hole in the bath is excentric to allow sufficient space for heating.

Under the diminished pressure caused by lowering the cistern, the water in the spindle soon boils vigorously, and, so far as my experience goes, without "bumping," and the expelled gases soon collect in the upper stem. After two hours' boiling, during which the process requires but little attention, the reservoir may be raised, the clamp (B) opened, the gases passed into the nitrometer, taking care not to let the "following water" rise as far as the capillary part of the spindle. The clamp is again closed, and the reservoir lowered, and the operation continued in order to see if any more gas appears. Finally the reservoir is raised, and the residual gas driven completely into the nitrometer, the "following water" being allowed this time to go as far as the nitrometer tap.

The apparatus is now disconnected from the nitrometer, the contents of which latter, after due interval, are subjected to measurement and absorption as usual.

The advantages claimed for the above apparatus are as follows: The water treated, together with the evolved gases, do not quit the vessel until the end of the operation, when the latter are delivered free from moisture into the measuring tube.

There are no corks, perforated or otherwise, and but few connections, while the latter are of a character admitting of being secured against leakage. The apparatus while working requires but little attention; so long as the water in the bath is kept boiling, that in the vessel may be regulated efficiently by due adjustment of the mercury cistern.

While this apparatus has rendered me excellent service in extracting the oxygen, nitrogen, and carbonic acid gas dissolved in water, I am of opinion that the *latter* gas, *per se*, can be more satisfactorily estimated by other methods, especially when chalk waters are operated upon, these latter retaining the last traces of free CO_2 with extreme tenacity even after prolonged boiling.

In conclusion, while it is possible that I may have been anticipated in the above contrivance, I feel it to be only fair to state that I am unaware of the fact, and should have been glad of its help long ago.

DISCUSSION.

The Chairman (Mr. Alfred H. Allen) thought Mr. Harvey had added a valuable method to those available for carrying out the troublesome operation of extracting gases from water. By operating under diminished pressure, the great advantage was obtained of securing the ready and complete removal of the gases, and Mr. Harvey had again shown the utility of the nitrometer, the use of which, for such purposes, he (Mr. Allen) was one of the first to advocate.

Mr. H. Droop Richmond asked Mr. Harvey how the free carbon dioxide compared with the combined carbon dioxide; were they equal in quantity, as they should be if the carbon dioxide existed as bi-carbonate? He (Mr. Richmond) had determined the free carbon dioxide, not by actual separate measurement, but by a very obvious method, that of adding an excess of standard baryta and titrating back, using

phenolphthalein as indicator. He had found in certain waters that there was a very considerable deficiency of free carbon dioxide, compared with the quantity that would be theoretically required to form hydro-carbonates. He might mention that in one or two cases the carbon dioxide obtained by the method that he had used had been compared with the actual carbon dioxide obtained by boiling the water, passing the steam through a condenser, absorbing and weighing the carbon dioxide. The combined carbon dioxide he had estimated by the method devised by Mr. Hefner for estimating hardness in water, that is, titrating the water with dilute standard acid, with methyl-orange as indicator.

Mr. Harvey, replying to Mr. Richmond, said that he was first attracted to the question of the accurate estimation of free carbon dioxide by the extraordinary discrepancies between his own results obtained, not by actual gas measurement, but by other methods, and those of other analysts who had previously examined the same samples, inasmuch as he had found 20 cubic inches per gallon in a certain water, and other observers had found 7 or 8. It was very difficult to get the whole of the gas out. Unless by very long boiling it was impossible, and in estimating he had given up the attempt to boil the water in an ordinary flask and at the ordinary pressure. In his opinion, the method he had shown was the only way in which the gas could be got out. By this means he had been able, for the first time, to confirm the results obtained through other processes. In chalk waters he had found the free CO_2 at least equal to the amount in combination with the lime present.

Dr. Teed asked Mr. Harvey if he paid any attention to the presence of sodium carbonate, as certain waters frequently contained that compound.

Mr. Harvey said that he did not find any sodium carbonate existing in such (chalk) waters. The sodium existed in the form of chlorides.

The Chairman said that he had known instances where 7 to 8 grains per gallon of carbonate of calcium had remained dissolved in the water, even after long boiling, provided that evaporation was prevented.

Mr. Harvey observed that in the precipitation of carbonate of lime by Clark's process there was a great deal to learn yet. He had watched the process day by day, and he was in a position to say that there were occasions when the precipitation was instantaneous, but it was within his knowledge that in some cases it was not complete for sixty or seventy hours afterwards.

The two following papers were read by Mr. Moor.

ON THE DETECTION OF EXHAUSTED GINGER.

By A. H. ALLEN AND C. G. MOOR.

SINCE the publication of the paper of Dr. Dyer and Mr. Gilbard in THE ANALYST for August, 1893, purchases of ground ginger under the Sale of Food and Drugs Act have been made in various parts of the country. In order to report on such samples to the best advantage, we have made a series of experiments on various specimens of ginger, and have collected data of considerable interest.

Taking advantage of the experience of Dyer and Gilbard, we have in each case determined the proportion of soluble ash, by which we mean the percentage on the original ginger which is dissolved out on treating the total ash with boiling water. In practice, perfectly good results are obtainable by regarding as the "soluble ash" the difference between the weight of the total ash and the ash insoluble in water. The aqueous solution can, of course, be used for determining the alkalinity, or the actual potash present.

Dyer and Gilbard found the proportion of soluble ash from genuine ginger to vary from 1.9 to 3 per cent., with an average of 2.7, while Mr. W. C. Young found in seven authenticated samples of ginger a range of 1.8 to 2.6 for the soluble ash. Figures for five samples of genuine ginger of unknown origin have been communicated to us by Mr. T. H. Pearmain, and show soluble ash ranging from 1.8 to 2.7 per cent., calculated on the moisture-free samples. The following are Mr. Pearmain's figures:

	1.	2.	3.	4.	5.
Total ash, less sand	3.1	3.9	3.7	5.0	4.5
Ash soluble in hot water	2.2	2.7	2.4	1.8	2.0
Fixed ether extract	3.2	3.0	2.5	5.0	4.2
Alcoholic extract after treatment with ether	2.7	3.1	3.4	2.9	3.0

These results bear out in the main those of Dyer and Gilbard.

Contrary to the general impression, for the manufacture of essence of ginger an aqueous or very dilute alcoholic liquid is employed. The use of any but very dilute spirit, 75° U.P., causes the solution of a considerable quantity of resinous matter, which is precipitated when the essence is subsequently diluted with the water of the ginger-beer. Hence, the use of alcohol in anything more than the most moderate proportion is neither necessary nor desirable, and the more so as the greater part of the pungent and flavouring principle of ginger is readily extracted by cold water. Exhausted ginger having already been subjected to treatment with water or a slightly alcoholic solvent, the soluble portions have been to a great extent removed, and hence such ginger yields a lower extract than a genuine article. The aqueous extract contains a considerable quantity of soluble salts of organic acids, and the ash is correspondingly deficient in soluble salts, and especially in compounds of potassium. In consequence of the presence in ginger of calcium salts soluble in dilute alcohol, the ash left on igniting the proof-spirit extract often considerably exceeds the proportion of soluble ash. This curious fact is no doubt due to the conversion of these calcium compounds into carbonate by ignition, thus rendering them insoluble in water.

In the hope of finding a solvent which would dissolve the valuable portion of the ginger without affecting the objectionable resinous matters, we have examined the action of cold water, proof-spirit, and rectified spirit on a number of samples of ground ginger of commerce. The following results were obtained by Mr. Haywood Court. Sample A was known to be genuine, but most of the others were purchased under the Sale of Food and Drugs Act, and some of them were ultimately admitted to contain an admixture of exhausted ginger.

	A.	B.	C.	D.	E.	F.	G.	H.	I.	J.	K.	L.	M.
Total ash	3.64	5.23	4.41	5.15	5.53	7.69	5.39	3.61	8.19	2.72	3.52	3.29	4.50
Ash soluble in hot water ...	2.36	2.59	2.22	2.57	2.87	2.36	—	1.24	1.45	0.69	1.11	0.97	1.37
Alkalinity of soluble ash as K ₂ O	0.96	0.96	0.29	0.13	0.15	0.20	—	0.27	0.20	0.23	0.23	—	—
Extracted by rectified spirit {	7.33	7.70	7.37	6.22	8.45	—	4.65	7.09	—	6.88	7.86	—	—
Extracted by proof spirit {	21.64	20.80	10.70	10.45	7.55	21.60	5.85	13.00	16.08	11.78	12.38	—	—
Containing ash	20.25	18.58	—	—	—	—	—	2.80	2.47	1.91	2.24	—	—
Extracted by cold water ...	14.57	13.16	14.95	14.55	14.50	14.60	8.14	8.33	9.78	8.51	7.18	8.08	7.39
Extracted by subsequent treatment with proof spirit	9.77	9.59	—	—	—	7.49	—	8.35	8.51	7.76	8.42	—	—
Extracted by subsequent treatment with rectified spirit	1.11	1.28	—	—	—	1.31	—	1.58	0.98	1.28	1.71	—	—
Total extract by three sol- vents used consecutively.	25.45	24.03	—	—	—	23.40	—	18.26	19.27	17.55	17.31	—	—

From these results it appears that the percentage of matter extracted either by proof or rectified spirit affords very little information, the large proportion of resinous matters yielded both by genuine and exhausted ginger masking any minor differences. It is true that Dyer and Gilbard consider that the proportion of matter extracted by alcohol after complete removal of the ethereal extract is a valuable item to the analyst; but as they found it to range from 2.1 to 3.8 per cent., while in the exhausted samples it varied from 0.8 to 1.4 per cent., it is evident that any estimate based on this datum must be of a very rough kind.

On the other hand, the proportion of extractive matter yielded to cold water seemed very encouraging, since the amounts dissolved in most cases followed closely the proportions of soluble ash. The following figures were obtained by Mr. R. Waterhouse by the analysis of genuine ginger of known origin, supplied to us by the kindness of Mr. W. Chattaway, of Apothecaries' Hall.

	N.	O.	P.	Q.	R.	S.	T.	U.	V.	W.	
Origin of Ginger.	Jamaica.	Jamaica.	Jamaica.	Jamaica.	Jamaica.	Cochin.	Cochin.	Cochin.	African.	African.	Average.
Moisture...	11.26	10.98	13.95	12.76	13.96	10.64	13.50	13.23	15.97	13.70	13.00
Total Ash..	—	—	3.90	3.29	3.45	—	3.81	3.62	3.66	3.90	3.66
Soluble Ash	1.70	1.41	3.05	1.75	1.71	1.71	2.03	2.04	2.28	2.41	2.01
Cold-water Extract.	15.65	13.25	14.40	12.25	11.85	13.00	8.65	11.65	10.80	10.10	12.12

Somewhat closer figures are obtainable by calculating the results on the moisture-free samples, but even then the figures show a good deal wider variation for the soluble ash than was observed by Dyer and Gilbard, while the cold-water extract also shows a wider range than we had expected. Sample *T* presents the peculiarity of yielding the average proportion of soluble ash, but an abnormally low water-extract. On the other hand, sample *O*, which gave the lowest soluble ash, yielded a cold-water extract above the average.

It appears, therefore, that neither the soluble ash nor the cold-water extract affords by itself a perfectly safe means of deciding as to the presence of exhausted

ginger, but by a combination of the two data it is possible to arrive at a more definite conclusion.

More extended observation is needed before anything like a definite limit of composition can be assigned to genuine ginger, and hence it is desirable to leave a wide margin when stating the proportion of exhausted ginger present in a sample. But, meanwhile, there is no difficulty in ascertaining the presence of the adulterant when it has been added in such quantities as to bring the soluble ash down to something like 1 per cent., and the cold-water extract to less than 8 per cent.; and this is the case with not a few gingers in the market. The adulteration has been admitted in several cases of this kind which have come under our notice.

We have pleasure in acknowledging the valuable assistance rendered by Mr. H. Court and Mr. R. Waterhouse in making the experiments above recorded.

DISCUSSION.

Dr. Bernard Dyer said that when he and Mr. Gilbard read their paper before the Society they dealt with Jamaica, Cochin, African and Bengal gingers. They had not at that time come across any samples of Japanese ginger. Since the reading of their paper he had analyzed three samples of Japanese ginger. They gave the following results:

		Approximate essential oil.		Fixed ether extract.		Alcoholic extract after ether.		Total ash (less sand).		Ash soluble in water.
"Japan"	0.60	...	4.12	...	1.96	...	5.15	...	1.66
"Limed Japan"	0.68	...	4.14	...	1.74	...	6.58	...	1.74
"Washed split Japan"	0.56	...	4.98	...	3.66	...	3.34	...	1.08

He did not know if Japanese ginger occurred to any extent in commerce.

Mr. Richmond, referring to the soluble ash, said that in the insoluble ash of milk he had observed the presence of a considerable quantity of alkaline salts, which possibly existed as sparingly soluble calcium, sodium, or potassium compounds. Such salts were also found in the ashes of many plants; for instance, he believed that several disputes had occurred with the analysts to the Board of Inland Revenue as to the amount of soluble ash in such things as tobacco and snuff, and in at least one of these cases discrepancies had been traced to the fact that calcium sodium carbonate was present. This was a very troublesome thing to wash out, and it was very hard to say when the operation was completely performed. He thought it probable that a small excess of calcium salts in ginger might keep back some of the soluble ash. In reality, determination of soluble ash was not a very accurate determination, and possibly, if, instead of estimating the soluble ash, the actual amount of alkaline base were estimated, more concordant figures would be the result.

Dr. Walter J. Sykes asked if the method of extracting and estimating the essential oil of ginger by means of petroleum ether, proposed by Dragendorff as a general method for the separation of essential oils from resins, had been tried. In the vast majority of substances the volatile oil of the plant-derivative was soluble in petroleum ether, and the resin insoluble.

Mr. W. Chattaway said that Japanese ginger was a thing almost unknown in the trade, so far as any large sale was concerned. He did not believe that split ginger

(which he had never met with) was used in commerce to any extent. He confirmed what Dr. Sykes had said respecting the direction in which it seemed most desirable to work in connection with the detection of exhausted ginger. The volatile oil figure in a sample of ginger would suffer from washing. A considerable quantity could be removed with cold water.

Mr. Allen said that it had been clearly shown that they were again dealing with one of those natural products which varied very materially in composition. Milk was supposed to be strictly a natural product, but in the case of ginger the substance was subjected to a certain treatment. He had become aware that it was usual to dip the ginger into boiling water as a preliminary stage in manufacture, and if it was dipped a little too much, or previously split, it was possible that more might be dissolved than analysts anticipated. It might be imagined that there was a sort of film of gelatinous starch formed on the outside of the root, and it would be difficult for the water to dissolve matter from the interior. They had tried to find some datum by which ginger could be judged. The aromatic principle of ginger was easily soluble in water, and although if ginger were soaked in water the flavour was not wholly removed, still a very great part of it went into the cold water, and it was only necessary to evaporate the water to discover that such was the case. It was highly probable, as suggested by Dr. Sykes, that it would be capable of being dissolved out by petroleum spirit, and might perhaps be extracted from its aqueous solution by agitation with that solvent. As to the estimation of actual alkalies mentioned by Mr. Richmond, it would be found in one of the tables that they did estimate the alkalinity of the soluble ash, and had expressed the results in terms of potash. He believed it was a fact that Mr. Stock estimated the potash as chloroplatinate, which seemed very desirable in certain cases. Although at present there was no single satisfactory datum on which to judge ginger, by a careful consideration of the results yielded by several methods of treatment it was possible to arrive at a very fair approximation to the truth. The descriptions of ginger referred to by Dr. Dyer were mere curiosities of no commercial interest, and hence the abnormal results yielded by them did not affect the practical value of the deductions from the proportion of soluble ash and other data. It was also an interesting fact that the gingers of low soluble ash had recently almost entirely disappeared from districts where prosecutions had been instituted.

ON THE CHANGE IN THE COMPOSITION OF BUTTER BY LONG KEEPING.

BY A. H. ALLEN AND C. G. MOOR.

In the discussion on a paper recently read before this Society by Mr. Richmond, a question arose as to the extent to which butter decomposed by long keeping. As a contribution to the existing knowledge on this obscure question, it occurred to us that it would be interesting to analyze some samples of butter which had been in our possession since November, 1888. These samples were closely packed in tins, and had remained intact since the packages were sealed in Denmark more than five

years ago. The butter-fat obtained by rendering the fresh butter was analyzed at the time very carefully, and the results were published in a paper by one of us in *THE ANALYST* for January, 1889.

Three tins of the "B" butter and one of the "O" butter still existed, and it is the fat from these which we have recently submitted to analysis. The samples of "B" were badly decomposed, but the one remaining portion of "O" showed little or no sign of decomposition. It will be observed that the change undergone by this butter, as indicated by the results of the analysis, is comparatively trifling, whereas in the samples of "B," in which the decomposition was much more profound, the changes are extreme. Further, the changes in portions "B 1," "B 2," and "B 3," which have been kept side by side since 1888, have not proceeded with the same rapidity.

We are pleased to acknowledge the zealous assistance of Mr. G. S. A. Caines, who made the recent analyses given in the following table :

	Butter marked "B."					Butter marked "O."		
	Rendered and Analyzed November, 1888.		Rendered and Analyzed March, 1894.			Rendered and Analyzed Nov., 1888.	Rendered and Analyzed Nov., 1888.	Rendered and Analyzed March, 1894.
	Copen- hagen.	London.	London. 1	London. 2	London. 3	Copen- hagen.	London.	London.
Specific gravity at 100° C.	·8639	·8640	·8634	·8696	·8730	·8641	·8641	
C.C. of $\frac{N}{10}$ alkali required for 5 grammes by Reichert-Wollny process ...	22·63	22·39	14·43	12·02	12·02	24·39	24·70	22·48
Percentage of KHO required for saponification ...	22·27	22·05	21·99	22·55	22·88	22·15	22·03	23·33
= Saponification equivalent ...	251·9	254·4	255·1	248·7	245·2	253·3	254·6	240·4
Soluble fatty acids, per cent. ...	—	$\begin{cases} 4·37 \\ 4·50 \\ 4·45 \end{cases}$	3·82	5·66	5·80	—	$\begin{cases} 4·60 \\ 4·66 \\ 4·77 \end{cases}$	5·89
Insoluble fatty acids, per cent. ...	—	$\begin{cases} 90·24 \\ 90·62 \end{cases}$	90·73	90·70	90·00	—	$\begin{cases} 89·90 \\ 90·30 \end{cases}$	85·78
Iodine absorption, per cent. ...	Not done	Not done	30·01	27·17	25·08	Not done	Not done	25·57
Condition of the butter when rendered ...	Fresh	Fresh	De- com- posed	De- com- posed	De- com- posed	Fresh	Fresh	Showed no signs of decom- position.

DISCUSSION.

Mr. Allen said that the figures were no doubt surprising, and he could not see his way to offer any definite explanation of them. It would be noticed how, in the

samples of "B" recently analyzed, the Reichert figure had fallen from 22 to 14, and even to 12, whereas in the sample marked "O" it had fallen from 24.7 to 22½. On the other hand, if the figures for the soluble acids were examined, it would be found that, out of three tins of the same butter kept side by side, only one had undergone decrease. The insoluble acids had not very greatly altered. The iodine absorption also varied. The figures seemed to indicate that it was impossible to tell from such data—without going into the differentiation of the nature of the volatile acids—what change had occurred; nor could it be said that, because one sample of butter had altered in a certain manner, another was likely to alter in the same way. The two samples "B" and "O" had behaved in a different manner, though they had been kept under the same conditions as to exposure, absence of light, etc. It was not very often that an opportunity presented itself of making analysis of butter after so long a period of time.

Dr. Sykes thought it possible that the differences observed on long keeping might be explained by the differing nature of the bacteria which had obtained access to the samples. So far as he knew, the effects of bacterial action upon fats had yet to be worked out.

Mr. Richmond said that, in the butters which had decomposed, the disturbance between the normal ratios of the figures determined was very marked. It would be noticed in the three samples "B" that the Reichert-Wollny figure had gone down very considerably. The figure for the soluble fatty acids had, in the first case, gone down a very little, but by no means in proportion to the volatile acids, and in the other two it had gone up. When butter decomposed a hydrolysis undoubtedly took place, and the liberated fatty acids would be partly dissolved in the water of the butter. The same order of things was apparent in the case of the butter "O" as in "B," although the Reichert-Wollny figure had not gone down to such a marked extent. The figure for the soluble fatty acids had, however, gone up. About two years ago he had occasion to make determinations on about fifteen or sixteen samples, and estimating the Reichert-Wollny figure and the soluble acids, he found that the relations between the two were fairly constant. A mean ratio of 86.5 per cent. was found in the distillate, and this agreed fairly with the ratios originally found by Mr. Allen. In the examples shown on the board it was very easy to see that the ratio of the quantity distilled over in the Reichert-Wollny process to the total soluble acid had gone down to something like 50 per cent. in the butter marked "B 1," and about 70 per cent. in the butters marked "B 2 and B 3." There was also a change in the iodine absorption. When the butters were analyzed in 1888 iodine absorptions were not determined. Working in Mr. Hehner's laboratory, he had also, through the kindness of Mr. Allen, examined these butters; he believed his Reichert-Wollny figure was almost the same; as far as his recollection went, the iodine absorption was close upon 40 per cent.; so taking this as the original figure, there had evidently been a large diminution. As regarded the increase in the soluble fatty acids, it was evident that they were not distilled over in the Reichert-Wollny process, and, therefore, that they could not be the same kind of acid as that distilled, that is to say, the lower fatty acids of the acetic acid series. Some years ago a series of papers was

published by Hazura, who studied the action of alkaline permanganate, and he obtained by oxidation hydroxyacids from the unsaturated fatty acids of various kinds. Some of these acids were soluble in water, and he (Mr. Richmond) thought it was highly probable that old butters would contain considerable percentages of hydroxyacids produced by oxidation. Hydroxyacids had a high density, and the increase of density again indicated the presence of this class of compound. He had in his possession a sample which had been sealed up by Dr. Vieth, and which had been kept for a considerable time; the air had been excluded from it, it had been kept in the dark, and it showed no signs of decomposition. It would seem that if the butter was excluded from the air it kept much better; and that oxidation was an important factor. This was one of the facts which led him to think that the soluble acids might be hydroxyacids soluble in water. From a study of the results of fractional distillation of butter acids he had evidence of a soluble acid of less volatility than butyric, and it was not impossible that this was a hydroxyacid (*e.g.*, lactic). He would like to mention that it was extremely useful to determine the rise of temperature with strong sulphuric acid. He had found in the case of olive-oil that the rise had a quantitative meaning, and that a certain amount of rise could be assigned to acids of each series. It would be extremely interesting to determine it if the authors had a sufficient quantity of the butters left. He (Mr. Richmond) had given up the use of 50 grammes of the oil or fat for 25 grammes oil and 5 c.c. of acid, and found the results more accurate. In fact, he found that, with the apparatus which he used, he could get within 1 or 2 tenths of a degree centigrade. Messrs. Allen and Moor's figures were of great interest, and there seemed considerable hope that the analysis of decomposed butter would afford information as to its original composition. Glycerol determinations might be of use, as probably this would be attacked.

Mr. Moor stated that in the case of the "B" butters the samples had attacked the tinned iron box, and were quite brown in colour; whereas this was not the case with the "O" butter, which did not show any appearance of decomposition, or possess the strong smell of bad cheese which characterized the "B" samples.

In the absence of the author, the following paper was read by Dr. Dyer :

CHESHIRE CHEESE.

BY CHARLES M. BLADES.

The cheese produced by the farmers of Cheshire at one time held the highest reputation of any obtained from the other counties in England, both for richness in flavour and in butter fat. This important article of food has, however, sadly deteriorated in quality, not through the loss of the art or skill in the manufacture, as it has frequently been asserted, or through the richness of the pastures declining, but through that pernicious habit of retaining a portion of the cream from the milk for butter making. By some the night's milk is skimmed, and the skimmed milk added to the morning milk to be converted into cheese, while the cream removed is made into butter. Others adopt the more scientific method of half milking the cows, and keep the first portion separate to be converted into cheese; the cows are then milked dry, and the latter portion used for making butter.

Much care and scientific attention have been devoted to check the skimming and watering of milk; surely the almost equally important article of food—cheese, which forms the staple diet of a very large portion of our population, demands more attention than it has hitherto received by the inspectors under the Food and Drugs Acts.

The following analyses represent samples obtained from respectable retailers, only one of which indicates whole-milk cheese:

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
Water ...	52.60	44.00	42.90	36.10	49.87	42.06
Fat ...	9.85	26.12	20.02	30.67	20.82	21.84
Casein, etc....	32.95	25.08	32.58	28.23	24.44	32.20
Ash ...	4.60	4.80	4.50	5.00	4.87	3.90
	100.00	100.00	100.00	100.00	100.00	100.00

DISCUSSION.

Mr. Allen thought that the results obtained by Mr. Blades were very curious. The amount of water struck him as being excessive. He did not think he had seen cheese with more than 35 or 40 per cent. of water. He would like to know how far Mr. Blades succeeded in completely extracting the fat from the cheese. Under ordinary circumstances he (Mr. Allen) had not troubled to get all the fat out. A convenient way was to grind up the cheese with sand prior to extraction. It was open to question whether Mr. Blades had succeeded in extracting the whole of the fat, and if he had not been successful in doing that it would modify the result.

Mr. Richmond, referring to the question of the estimation of the fat, said that there was a method used by the American Association of Official Agricultural Analysts for the estimation of fat in cheese, which consisted in weighing out a portion of the cheese and grinding it up with twice its weight of anhydrous cupric sulphate, which absorbed the whole of the water, and the fat was comparatively easily extracted. From the results reported by them the method seemed to give exceedingly concordant results. He wished to know whether any information had been given in the paper as to how the casein was estimated. He presumed it was either estimated by determining the total nitrogen and multiplying by a factor, or by a subtraction of the weight of the water, fat and ash. Either method would give too high results for casein. Personally, he held the view that there was no casein in cheese. The albuminoid matter in cheese was the product of the action of rennet, and it contained a sensibly smaller proportion of nitrogen than casein. Casein contained about 15.7 per cent. of nitrogen, while the albuminoid matter of cheese contained about 14.3 per cent. In estimating the casein or the albuminoid matter of cheese by the estimation of nitrogen it was necessary to multiply by a higher factor than 6.25 or 6.33, or 6.38, which were the factors generally used. In the ripening of cheese there was a considerable amount of decomposition of the albuminoid matter. Small quantities of amido-compounds, and even ammonia, were found, and, in estimating the nitrogen of cheese, those ought to be taken into account. He thought that the only way of actually estimating the albuminoid matter of cheese was to form an insoluble

albuminoid compound by means of acetate of lead, or by Stutzer's method, and wash the other compounds out, estimating the nitrogen, and assuming that there was 14.3 or so in the cheese. In making cheese, when the milk was submitted to the action of rennet, the whole of the albuminoids were not contained in the cheese obtained—there was a considerable percentage in the whey. If it was assumed that milk contained 3.4 per cent. of albuminoid matter, the amount which went into the cheese was about 2.7 per cent. The greater portion of the fat was taken down, too; but there was also some fat left in the whey. He thought the average left was nearly 1 per cent., though this varied considerably. In his experience of the last year, the exceptional weather which had prevailed had had more effect on the quality of Cheshire milk than it had on the quality of the other milks which he had examined. It was true these milks came from half a dozen farms, and he could not say that the samples were strictly authenticated in the legal sense, but there were peculiar circumstances about them which led him to think that the milks had not been adulterated. If his experience were true for the whole of the county, it would account for the fact that the quality of Cheshire cheese was not as good as it has been. The proportions of fat to casein, 26 to 25, 31 to 28, 21 to 24, and possibly even 21 to 32 (that is, after making some allowance for the way in which the casein was estimated) were not so extremely low in fat considering the poor quality of the milk in the district during the past season. With regard to the first one, which only contained 10 of fat to 37 of casein, he thought that was distinctly low, and there was not any doubt at all that it was a cheese made from skimmed milk. The third and fifth might also be partially made from separated milk; but as for the others, he thought they were all of them within the limits of the results which would be obtained from the analysis of cheese made from poor milk. He could not agree that the analysis of the cheeses given by the author showed distinct evidence of a general skimming of the milk, and, from what he knew of Cheshire farmers, thought that the falling off in the quality of the cheese was due to the milk having been exceptionally poor.

NOTE ON THE ANALYSIS OF PHOSPHOR TIN.

By FRANK L. TEED, D.Sc.

(Read at the Meeting, April 4, 1894.)

The ordinary method of analyzing this substance is by acting on it with nitric acid, evaporating to dryness, fusing with alkaline carbonates, and extracting the fused mass. Another method is to fuse the finely-divided substance with a mixture of alkaline carbonates and nitrates, and extract the fused mass.

Both these methods have the objection of a fusion being necessary, and in all accurate work a fusion should be avoided if possible. The method I venture to recommend is to oxidize with nitric acid, destroy the excess of nitric acid with ammonium chloride, make alkaline with ammonia, and then warm with excess of ammonium sulphide until complete solution of the tin. Any traces of metals whose sulphides are insoluble in ammonium sulphide are here left undissolved, and may be filtered off.

On acidifying this ammonium sulphide solution the Stannic sulphide is precipitated, and the phosphorus is estimated in the filtrate by addition of ammonia and "magnesia mixture."

DISCUSSION.

Dr. Bernard Dyer said that in the analysis of phosphor tin, he had not been successful in separating the tin and the phosphoric acid in the way that Dr. Teed had suggested, although he had tried it. He found that the phosphoric acid was retained by the tin when the latter was precipitated as sulphide, despite the acidity of the solution. It was an exceedingly difficult thing to separate tin and phosphoric acid. He thought the most accurate way to determine the phosphorus in phosphor tin was the following: First attack the metal with nitric acid, subsequently add hydrochloric acid, and thus get all the phosphorus into the state of phosphoric acid and the tin into solution. The hydrochloric acid should be present in as small a quantity as possible, only just enough to ensure the metal being kept in solution. This was important. Then the phosphoric acid could be determined in the usual way by means of molybdenum. Of course it was easy to dissolve the phosphor tin in hydrochloric acid at once, but that involved a wholesale loss of phosphorus in the form of hydrogen phosphide. He had tried Dr. Teed's method—or, rather, the similar method laid down in Fresenius—some two or three years ago, and though he made several attempts, he could not make it work satisfactorily.

Mr. Hehner said he would like to know how Dr. Dyer had treated his precipitate. The precipitate should consist of oxide of tin.

Dr. Teed inquired if Dr. Dyer had been very careful in getting rid of his nitric acid; if that were not properly effected the phosphoric acid must go down.

Dr. Dyer said it was some time since he performed the experiments, and he could not remember the details; but he followed those laid down by Fresenius, and was quite disappointed with the result.

NOTE ON LEMON AND ORANGE PEEL.

By E. G. CLAYTON.

(Read at the Meeting, April 4, 1894.)

When orange-peel is moistened with strong hydrochloric acid, its colour changes from yellow to a rich dark green; lemon-rind, similarly treated, retains its hue, or, at most, assumes a dingy, yellowish-brown tint. A convenient and simple chemical test, therefore, which will distinguish between small fragments of lemon and orange peel is to touch them with a glass rod previously dipped in hydrochloric acid. The diluted acid will answer the purpose, but the reaction is slower.

A few minutes' exposure to hydrochloric acid gas will effect this change in the pigment of orange-peel. The colour of lemon-rind is unaffected.

The shades of green developed by dilute hydrochloric acid are deepest in the cases of Mercia, Denia, and Florida oranges; of moderate intensity with Jaffa and

"blood" oranges; and feeble with Valencia and Tangerine oranges. This statement also applies to the reactions with *strong* hydrochloric acid, excepting that the colour of Tangerine orange-peel with the strong acid is perhaps more intense than that observed with any of the other varieties of the fruit.

The peel of the lime behaves, with hydrochloric acid, like lemon-rind.

Mr. Hehner remarked that it was not at all an infrequent occurrence for lemon and other peels to be artificially coloured. He thought that at present too implicit reliance should not be placed on this test in the case of candied-peel.

The Determination of Phosphoric Acid by the Titration of the Yellow Precipitate with Standard Alkali. H. Pemberton, Jur. (*Jour. Am. Chem. Soc.*, 1893, 382.)—In a paper read in 1882 the author called attention to the satisfactory results yielded by this method, but it is only recently that he has been able to work the subject out. Since 1882 several chemists have proposed processes based on the same principle, their names and a short description of each modification being given.

In the author's process the following solutions are employed:

Ammonium Molybdate.—Ninety grammes of the crystals are dissolved (in a large beaker) in somewhat less than one litre of water. This is allowed to settle overnight, and the clear liquor decanted into a litre flask. The small quantity of insoluble molybdic acid, always present, is dissolved in a little ammonia and added to the main solution. Should the molybdate be found to contain traces of P_2O_5 , a few decigrammes of magnesium sulphate are added, ammonia being added to faint alkalinity. The whole is then made up to one litre. It is this *aqueous* solution that is used, *no nitric acid whatever being employed*. Each c.c. precipitates 3 milligrammes of P_2O_5 .

The *ammonium nitrate* solution is simply a saturated aqueous solution of the salt. Distilled water is poured into the bottle of crystals in quantity insufficient to dissolve them all. Even in cold weather 10 c.c. of this solution is amply sufficient for each test.

The *nitric acid*, used for acidifying the solution of the phosphate, has a specific gravity of 1.4 or thereabouts.

The *standard potassium hydroxide* solution is of such strength that 1 c.c. = 1 mgm. P_2O_5 . One hundred c.c. of it will neutralize 32.65 c.c. of normal acid. It can be made from normal potassium hydroxide (that has been freed from all carbonate by barium hydroxide) by diluting 326.5 c.c. (see next paragraph) to 1 litre. But its strength is best determined empirically by a direct test upon a phosphate solution of known strength, precipitating with ammonium molybdate, and making the analysis as described below, all potassium carbonate having first been removed by barium hydroxide.

In a subsequent paper (*Jour. Am. Chem. Soc.*, 1894, 278), the author gives the details of a number of experiments which he had made, proving that 23 molecules of NaHO are required to neutralize the yellow precipitate containing 1 molecule of

P_2O_5 , and not 23.2 as his previous experiments had led him to conclude. Consequently, the above-mentioned *standard potassium hydroxide* solution should be made by diluting 323.7 c.c. normal solution to a litre instead of 326.5 c.c.

The *standard acid* has the same strength, volume for volume, as the potassium hydroxide, and can be made by diluting 323.7 c.c. of normal acid to 1 litre. In testing it against the alkali, phenolphthalein (and methyl orange) should be used.

The *indicator* can be either litmus, rosolic acid, or phenolphthalein. I have used the latter almost exclusively, as it has been shown by J. H. Long (*Am. Chem. J.*, xi., 84) that titrations with this indicator in the presence of ammonium salts are perfectly reliable if the amount of the ammonium salt present is not excessive, if the solution is cold, and if the phenolphthalein is used in sufficient quantity. One gramme of the phenolphthalein is accordingly dissolved in 100 c.c. of 60 per cent. alcohol, and, at least, 0.5 c.c. of this solution is used for each titration. The washing of the ammonium phospho-molybdate is done by water. (Isbert and Stutzer, *Ztschr. anal. Chem.*, xxvi., 584, have shown that there is no danger of loss in washing the yellow precipitate with water.)

The following is the method of performing the analysis:

One gramme of the phosphate is dissolved in nitric acid, an excess of which can be used with impunity, and the solution filtered into a 250 c.c. flask and made up to the mark. The solution can even be poured into the flask without filtering, since the presence of a little insoluble matter does not interfere in the least with the titration. Moreover, since most phosphate rocks seldom contain over 10 per cent. of insoluble matter, and as this has the specific gravity of, at least, 2, it occupies a volume of about 0.05 c.c., an amount so small that it may be neglected. (For instance, even in the case of a phosphate rock containing 40 per cent. P_2O_5 , the error is only 0.008 per cent. P_2O_5 .)

After the clear solution has been poured off, it is well to treat the sand, etc., at the bottom of the beaker, with a c.c. or so of hydrochloric acid, in the warmth, to insure complete solution.

It is not necessary to evaporate to dryness. Isbert and Stutzer have shown in their paper that when the yellow precipitate is washed with water, the soluble silica is removed, and that evaporation (to render the silica insoluble) is superfluous. Their results have been corroborated by test analyses. In the event of its being desirable to remove silica by evaporation for any purpose, the evaporation should be performed over a water-bath, or, if on an iron plate, with great care, since, otherwise, meta- or pyro-phosphates are formed, with results that are correspondingly low.

Twenty-five c.c. of the solution (equal to 0.1 gramme) are now measured out and delivered into a beaker holding not more than 100 to 125 c.c. A large beaker requires unnecessary washing to remove the free acid in washing the yellow precipitate. The solution is neutralized with ammonia—until a precipitate just begins to form—and 5 c.c. of nitric acid of sp. gr. 1.4 added; 10 c.c. of the ammonium nitrate solution are added, and the entire bulk of the solution made up to 50 to 75 c.c. by adding water.

Heat is now applied, and the solution brought to a full boil. It is then removed from the lamp, no more heat being applied, and treated *at once* with 5 c.c. of the

aqueous solution of ammonium molybdate, which is run into it from a 5 c.c. volume pipette, the solution being stirred as the precipitate is added. The beaker is now allowed to rest quietly for about one minute, during which time the precipitate settles almost completely. The 5 c.c. pipette is filled with the molybdate solution, and a part of its contents allowed to drop in, holding the beaker up to the light. If a formation of a yellow cloud takes place—it is at once perceptible—in which case the remainder of the pipetteful is run in, the solution stirred and allowed to settle. A third pipetteful is now added as before. Should it cause no further cloud, only about one-half of its contents is added, the remainder being run into the beaker into which the filtrate and washings from the yellow precipitate are to go. In test analyses it was shown that, even when 15 c.c. in excess of the molybdate were purposely used over and above the calculated amount, the results were accurate, no molybdic acid coming down with the yellow precipitate.

It is seldom that more than 15 c.c. in all (three 5 c.c. pipettefuls) of the molybdate have to be added. Since each c.c. precipitates 3 milligrammes P_2O_5 , 15 c.c. will precipitate 45 milligrammes P_2O_5 . This is equivalent to 45 per cent. on the 0.1 gramme taken for analysis, and it is not often that any material to be examined contains over this percentage. In the analysis of materials rich in phosphoric acid, it is one of the embarrassing features of the usual process, in which the *nitric acid solution* of the molybdate is used, that, in the first place, large quantities of the precipitant have to be used (frequently several hundred c.c.), and in the second place, that the analyst is never certain that enough has been added to throw down all of the phosphoric acid. This necessitates frequent testings of small portions of the phosphate solution or of the filtrate. There is another difficulty peculiar to the process as usually carried out in all methods in which the determination is made directly upon the phospho-molybdate itself, in that much care must be observed to keep the solution at a certain temperature, since otherwise molybdic acid contaminates the precipitate and the analysis is rendered worthless. In the process herein described, using an *aqueous* solution of the molybdate, the point at which sufficient of the precipitant has been added is easily seen. No molybdic acid separates, because, in the first place, no great excess of molybdate is added; and because, in the second place, the solution is filtered immediately, or as soon as it has settled, which requires only a minute or two. The time required from the first addition of the molybdate to the beginning of the filtration is never over ten minutes, and is generally less. The filtrate and washings from the precipitate when treated with additional molybdate solution, give, on standing on a hot plate for an hour or so, a snow-white precipitate of molybdic acid, showing that all of the phosphoric acid has been precipitated.

A slight correction should be made to the statement made above in regard to 15 c.c. of the molybdate precipitating 45 milligrammes of P_2O_5 . This is not strictly true, for the reason that a small quantity (something over 1 c.c.) of the molybdate is required to neutralize the solvent action of the nitric acid. Therefore, in *very* high grade phosphates a fourth 5 c.c. pipetteful may be required.

The yellow precipitate is now filtered through a filter 7 centimetres in diameter, decanting the clear solution only. This is repeated three or four times, washing

down the sides of the beaker, stirring up the precipitate, and washing the filter and sides of the funnel above the filter each time. The precipitate is then transferred to the filter and washed there. When the precipitate is large it cannot be churned up by the wash water, and cannot be washed down to the apex of the filter. This is generally the case when there is over 10 or 15 per cent. phosphoric acid present in the substance analyzed. In such an event, the precipitate is washed back into the beaker, and the funnel filled with water above the level of the filter, this being done two or three times, then the precipitate washed back into the filter. It is not necessary to transfer to the filter the precipitate adhering to the sides of the beaker.

It goes without saying that during the washing no ammonia must be present in the atmosphere of the laboratory. Inasmuch as the beaker, funnel, filter and precipitate are small, the washing does not take long to perform. It requires, in fact, from ten to fifteen minutes, even when large precipitates ($= 30$ to 40 per cent. P_2O_5) are handled. The precipitate and filter are now transferred together to the beaker. The alkali solution is run in until the precipitate has dissolved, at least twelve drops of the phenolphthalein ($1 : 100$) are then added, and the acid run in without delay until the pearly colour disappears and the solution is colourless. The presence of the filter paper does not interfere in the least. The reaction of the indicator is not so sharp as when only acid and alkali are used, but it is easy to tell with certainty the difference caused by one drop of either acid or alkali. After deducting the volume of acid used from that of the alkali, the remainder gives the percentage of P_2O_5 directly, each c.c. being equal to 1 per cent. P_2O_5 . Thus, if there are 28.3 c.c. of alkali consumed, the material contains 28.3 per cent. P_2O_5 , when one decigramme is taken for analysis. From the time the 25 c.c. are measured out until the result is obtained, from thirty to forty minutes are required.

The author has applied this process to determinations of phosphoric acid in phosphates and fertilizers, but has had no experience in determining phosphorus in iron, steel, or iron ores. He is inclined to believe that in the presence of such large quantities of iron salts, when using the aqueous solution of the molybdate, it may be necessary to guard against contamination of the yellow precipitate by ferric hydrate, perhaps by using larger quantities of nitric acid than 5 c.c., and perhaps by washing the precipitate at first with dilute nitric acid. It may also be the case that the yellow precipitate will form more slowly.

Test analyses gave satisfactory results.

W. J. S.

The Volumetric Estimation of Sulphuric Acid. W. Windisch. (*Wochen-schrift f. Brauerei*, 1894, p. 607.) This method depends upon the precipitation of the sulphuric acid by an excess of barium chloride solution of known strength; the excess of barium is next removed by an excess of potassium chromate solution (also of known strength), and then the excess of chromate quantitatively determined. Several methods were tried for the last determination, first oxalic acid, then ferrous salts, both of which were rejected, since it was found that the former acted irregularly, and that it was difficult to secure stable solutions of the latter. Arsenious acid, which acts according to the following equation, $4CrO_3 + 3As_2O_3 = 2Cr_2O_3 + 3As_2O_5$, proved a

reliable agent for this purpose, and it was shown that the reaction was the same either in an acid or an alkaline medium.

The following decinormal solutions are required : (1) Solution of barium chloride, containing 23.86 grammes of the salt per litre ; (2) potassium chromate solution, with 19.4561 grammes per litre ; (3) arsenious acid solution, with 4.95 grammes As_2O_3 per litre ; (4) iodine solution, containing 12.7 grammes per litre. The arsenious acid, which must be perfectly pure, is dissolved in a small quantity of warm NaOH solution, and then sulphuric acid added to slight acid reaction.

In the actual determination $\frac{N}{10}$ solutions, prepared by diluting the above, are made, and these are titrated the one against the other, in order to ensure absolutely accurate relations between them.

For the estimation of the sulphuric acid in a water, 100 c.c. of it are taken ; or, in the case of waters containing small quantities of sulphates, several 100 c.c. are evaporated to 100 c.c., with the addition of a little HCl , the water being subsequently neutralized, or made slightly alkaline with ammonia. The water is brought to the boil, 50 c.c. of the $\frac{N}{10}$ barium chloride added, and immediately afterwards an equal quantity of the $\frac{N}{10}$ potassium chromate solution. After being boiled for one or two minutes, the whole is brought into a 300 c.c. flask, cooled and made up to 300 c.c. Portions of 100 c.c. each are now filtered off, and to each of these 50 c.c. of the $\frac{N}{10}$ arsenious acid solution and 5 c.c. of 20 per cent. H_2SO_4 added, well shaken, and allowed to stand until the solution has become colourless : this takes place in two or three minutes. Should the first 50 c.c. not be sufficient to decolorize the mixture, a second similar quantity is added. A little starch solution is then added, also a small quantity of a cold saturated solution of hydric sodium carbonate, and the liquid titrated with the $\frac{N}{10}$ iodine solution, until a permanent pale-blue colour is produced. If T equals the titre between the iodine and arsenious acid solutions, T_2 that between the arsenious acid and potassium chromate solutions, and I the number of c.c.'s of iodine solution used, then : $x = \frac{12 (T_1 - I)}{T_1 \times T_2}$ grammes of sulphuric anhydride (SO_3) in the quantity of the water taken.

A number of examples are given showing that the process gives results which are in the most satisfactory concordance with those of the ordinary gravimetric process, both in the estimation of the H_2SO_4 in waters and also in sulphates. In estimating the H_2SO_4 in the sulphates of the heavy metals, the metallic oxide is first removed by NH_3 , the solution being afterwards neutralized with HCl . Raw gypsum is first fused with dry sodium carbonate, the melt dissolved in dilute HCl , and exactly neutralized with NH_3 .

W. J. S.

The Tannin-Bodies in Hops, and their Estimation. M. Hayduck. (*Wochens. f. Brauerei*, 1894, p. 409.) The author has prepared samples of hop-tannin and of hop-phlobaphene by Etti's method (*Annalen d. Chemie*, 1876, vol. clxxx.). He describes the pure hop-tannin as being a bright-brown amorphous powder, soluble in water, acetic ether and dilute alcohol ; very slightly soluble in absolute alcohol, and insoluble in ether. The 0.2 per cent. aqueous solution has a pale-yellow colour, and shows a greenish fluorescence. Its taste is at first slightly bitter, afterwards astringent.

gent, though not unpleasantly so. Fe_2Cl_6 produces an intense green coloration, but no precipitate; and this forms an exceedingly sensitive test for the presence of hop-tannin. In accordance with Etti's statements, Hayduck found that hop-tannin is precipitated by albumin, but not by gelatin; but, contrary to Etti's statement, it was found to be precipitated by animal membrane. Hop-tannin is acid to litmus; it is decomposed with remarkable ease, simple evaporation in aqueous solution being sufficient to partially convert it into a substance insoluble in water, a small portion being also transformed into phlobaphene. Its complete conversion into phlobaphene is readily effected by the agency of alkalies. If a solution of the tannin be made alkaline with sodium carbonate and evaporated, complete conversion ensues; the same also happens if the tannin be heated in the dry condition to 140°C .

The phlobaphene is a reddish-brown powder, partly soluble in boiling water or dilute alcohol. Its aqueous solution has a yellowish-brown colour, a disagreeable bitter, astringent taste, and gives a dirty dark-green precipitate with Fe_2Cl_6 ; it is precipitated by albumin, and is also removed from its aqueous solution by hide. The larger portion of the phlobaphene obtained is insoluble in water, but dissolves in alkaline solutions with a deep-brown colour, from whence it is reprecipitated by acids. The soluble form shows a great tendency to pass into the insoluble one, simple boiling in water, or heating the dry substance to 130°C ., effecting this change.

After reviewing the various methods which have been proposed from time to time for the estimation of the tannin-bodies in hops, Hayduck concludes that Lowenthal's method is the most reliable one, and proposes the following procedure: The hops are first extracted with ether to remove hop-resin, dried, and coarsely powdered in a coffee-mill. Ten grammes of the hops so prepared are extracted by boiling with six successive portions of distilled water of 100 c.c. each. The first boiling lasts an hour, the subsequent ones half an hour each. The extracts so obtained are mixed and made up to 500 c.c., cooled and filtered. An apparatus designed by Prof. von Schröder is strongly recommended for the extraction. It consists of a cylindrical vessel of tin-plate, provided with a spout, into which fits fairly tightly a perforated plate, furnished with a handle. The perforated plate is covered with fine gauze, and after each boiling the plate is gently pressed down towards the bottom of the vessel; the solid matter is retained by the strainer; the extract passes through the gauze. To remove the hop-tannin-bodies from the portion of the solution required for the second titration, 5 grammes of powdered hide are soaked in water for twenty-four hours, squeezed as dry as possible in a linen cloth, and added to 100 c.c. of the hop-extract, then allowed to remain for twenty-four hours, the mixture being occasionally well shaken. The quantity of water taken up by the powdered hide is estimated and allowed for subsequently.

For the actual determination 10 c.c. of the original hop-extract and 10 c.c. of indigo solution are taken, diluted with water to 750 c.c., and titrated with permanganate in the usual manner. The same quantity of the extract after treatment with the hide powder is similarly titrated. The following are the results of such a determination:

Titre of permanganate, 1 c.c. = 0.002026 gramme tannin.

Permanganate required to decolorize 10 c.c. indigo solution = 9.4 c.c.

The 5 grammes of powdered hide had absorbed 11 grammes of water.

The hops contained 9.92 per cent. moisture.

Titration of 10 c.c. of original hop-extract = 12.35 c.c.

Titration of 10 c.c. of hop-extract after treatment with powdered hide = 10.05 c.c.

Deducting from each of these figures the 9.4 c.c. used for decolorizing the indigo, leaves 2.95 c.c. and 0.65 c.c. respectively; and this latter figure, after allowing for the water absorbed by the hide-powder, becomes 0.72. Then $2.95 \text{ c.c.} - 0.72 \text{ c.c.} = 2.23 \text{ c.c.}$ permanganate used by the tannin-bodies, which is equivalent to 2.258 per cent. tannin-bodies in the original hops, or 2.50 per cent. in the water-free hops.

The results obtained in comparative experiments, made with hops before and after treatment with ether, showed very small differences; they were slightly higher (about 0.25 per cent.) in hops without previous treatment. The author considers that it is a difficult matter to decide which of the two is the more correct figure. He has ascertained that the presence of hop-resin in the solution does not make any appreciable difference in the result, and conjectures that the treatment with boiling ether in the extraction apparatus may cause a portion of the tannin-bodies to become converted into the insoluble form.

Some very valuable observations on the part played by hops in brewing conclude the paper.

W. J. S.

A New Method of Analyzing Fats and Resins. Parker C. McIlhiney. (*Jour. Am. Chem. Soc.*, 1894, p. 275.) A number of processes for the analysis of fats, depending upon the power possessed by their unsaturated constituents to absorb by direct addition two or four atoms of bromine or iodine, have been proposed and used, amongst others, by Allen (*Analyst*, vi., 177); Mills and Snodgrass (*Jour. Soc. Chem. Ind.*, ii., 436); Mills and Akitt (*Jour. Soc. Chem. Ind.*, iii., 65); Hübl (*Dingler's Poly. Jour.*, ccliii., 281, and *Jour. Soc. Chem. Ind.*, iii., 641); Levallois (*Jour. pharm. Chim.*, 1887, i., 334); Halphen (*Jour. pharm. Chim.*, 1889, xx., 247); Gantter (*Ztschr. anal. Chem.*, 1893, 178).

The aim of all these processes is to determine the amount of halogen which the substance under examination will absorb by addition, but the figures obtained represent this only approximately even when substances which easily form substitution products are absent. Some substitution takes place with almost all oils, and with rosin-oil, rosin, and probably most other resins, substitution causes the entire absorption.

The extent to which this substitution takes place depends upon the nature of the substance operated upon, and varies with different oils and resins, and a determination of the amount of halogen so absorbed may serve as a means of identifying and in some cases determining them.

The following process has been devised for determining the amount of bromine which oils and resins can absorb by addition (which will be called the "Bromine Addition Figure"), and at the same time the amount of bromine which replaces hydrogen, the action being allowed to continue eighteen hours in the dark; this gives the "Bromine Substitution Figure." The first figure gives in most cases the same information as the Hübl figure, but is more reliable, while the second figure is a measure of the activity of the saturated constituents toward bromine.

It depends upon the fact that bromine, in forming substitution compounds, forms a molecule of hydrobromic acid for every atom of bromine which replaces hydrogen, while in forming addition compounds no hydrobromic acid is formed.

It was found impossible to use iodine alone, as the addition figures are then very much too low, and there is little difference between the substitution figures of bodies of unlike character.

The following solutions are used :

Bromine in carbon tetrachloride	$\frac{N}{33}$
Sodium thiosulphate	$\frac{N}{10}$
Potassium hydroxide	$\frac{N}{10}$

0.250—1.000 gram of the substance is dissolved in 10 c.c. of carbon tetrachloride in a bottle of 500 c.c. capacity provided with a carefully ground glass stopper. An excess of bromine solution is added, the bottle tightly stoppered and placed in a dark closet. No water or alcohol should be present, and light should be excluded as far as practicable. At the end of eighteen hours the bottle is cooled with ice to form a partial vacuum, and a piece of wide rubber tubing about one and one-half inches long is slipped over the lip of the bottle so as to form a well about the stopper. This well is filled with water and the stopper carefully lifted, when the water will be sucked into the bottle and dissolve the hydrobromic acid present. When about 25 c.c. of water have been added in this way, the bottle is well shaken, and 10—20 c.c. of 20 per cent. potassium iodide solution added. The excess of bromine acts on the potassium iodide, liberating a corresponding amount of iodine which is titrated with $\frac{N}{10}$ thiosulphate after adding about 75 c.c. more water, using starch as an indicator. The total bromine absorption is calculated from the difference between the amount of thiosulphate required for the bromine solution added and the amount required for the excess. The contents of the bottle are now transferred to a separatory funnel and the aqueous portion separated, filtered through a cloth filter, a few drops of thiosulphate added if the solution is blue, and this is then titrated with $\frac{N}{10}$ potassium hydroxide, using methyl orange as indicator. The end reaction is best observed by using a porcelain casserole to contain the solution, adding the alkali in slight excess and titrating back with $\frac{N}{10}$ hydrochloric acid until the pink acid tint just reappears. From the number of cubic centimetres of alkali used the amount of bromine present as hydrobromic acid is calculated, and when expressed in per cent. gives the bromine substitution figure, because for every atom of bromine which has replaced an atom of hydrogen, one molecule of hydrobromic acid has been formed. Twice the bromine substitution figure subtracted from the total absorption gives the bromine addition figure.

The following results were obtained :

Substance.	Total bromine absorption, eighteen hours.	Bromine, addition figure.	Bromine, substitution figure.
W. G. Rosin ...	212.7	0.0	106.35
E. Rosin ...	206.5	0.0	103.25
Second run Rosin Oil (a)	116.2	0.0	58.1
" " " " (b)	114.7	0.0	57.35
American Raw Linseed Oil	102.88	102.88	0.0
Same Oil Boiled	103.92	103.92	0.0
White Salad Cotton-seed Oil	65.54	64.26	0.64
Sperm Oil	56.60	54.52	1.04

A consideration of the above figures shows that the results are much more instructive than those obtained by the Hübl process which is the one in common use. Rosin-oil, rosin, and other resins may be detected and determined in mixture with fatty oils, or, if they are present in known quantity, the character of the fatty oil may be determined. Investigations which are being made on a large number of oils and resins will probably furnish analytical data for the analysis of oils and varnishes.

W. J. S.

An improved Volumetric Precipitation Process. P. N. Raikow. (*Chem. Zeit.*, 1894, xviii, 484, 485.)—The cardinal fault of most volumetric precipitation processes is that no direct reading of the end-point is possible, filtration and trial of small portions of the clear filtrate being usually necessary. The author has found that many precipitates which remain obstinately suspended under ordinary conditions, and cause in the liquid being titrated an unmanageable turbidity, can be induced to collect and subside by the addition of some immiscible liquid heavier than water, e.g., carbon disulphide or chloroform. Such liquids, although exerting no solvent action on the precipitate, mix intimately with it and carry it down, leaving the supernatant liquid sufficiently clear for the observation of any turbidity produced by the addition of a further quantity of the standard precipitating solution. Carbon disulphide and chloroform are usually, but not invariably, effective. Thus carbon disulphide carries down silver chloride rapidly and completely, but has no influence on the precipitation of barium sulphate. The application to particular cases of the principle here enunciated is being worked out by the author.

B. B.

REVIEW.

ANIMAL AND VEGETABLE FIXED OILS, FATS, BUTTERS, AND WAXES: THEIR PREPARATION AND PROPERTIES, AND THE MANUFACTURE THEREFROM OF CANDLES, SOAPS, AND OTHER PRODUCTS. By C. R. ALDER WRIGHT, D.Sc., B.Sc., F.R.S., with 144 illustrations. Price 28s. (London: Charles Griffin and Company, Limited.)

This work is one of those which almost defy the reviewer. The vast amount of detail, the numerous tables, and the breadth of the subject, render any concise description of the work difficult, if not impossible.

In his preface, the author admits that the complete discussion of the sources, production, and general technology of the numerous substances included in the term "oils" would require far more space than is compatible with the limits of the work, and hence it has been found indispensable to make a selection, with the result that the subjects are now narrowed down to animal and vegetable fixed oils and allied substances, whilst mineral oils, products of distillation, essential oils, and various analogous materials are only discussed in so far as they are associated with the fixed oils in their technological application. In short, the object aimed at has been rather to give general descriptions of the methods whereby animal and vegetable oils and fat are obtained from natural sources, of their leading practical applications and uses, and of their chief physical and

chemical properties and reactions, than to enter into special details, and to discuss minutely the analytical tests and processes applicable in each separate case for the detection of adulteration.

The author proceeds to express his indebtedness to various scientific and technical journals, and in particular to the works of Schädler, Allen, and Benedikt. The author has culled extensively, yet with discretion, from the treatises of these authors, and with due acknowledgment of the source of his information. Whether the authors of these treatises will appreciate the wholesale conveyance of their tables and subject-matter to Dr. Wright's work is very doubtful, but there can be no question that the readers of the book will be greatly the gainers thereby.

Dr. Wright's compilation appears to be exhaustive, and the result is that his work will be found absolutely indispensable by every chemist interested in the subject of oil and fat analysis. It is refreshing to find a work on technical chemistry edited in a thoroughly scientific manner, and free from the slipshod chemistry which distinguishes some of the more pretentious works recently published.

After treating of the sources and general nature of fixed oils, Dr. Wright proceeds to describe the products of their saponification. Section II. deals with the physical properties of oils, fats and waxes, and the various tests for their purity and identity based thereon. In Section III. the chemical properties of oils, etc., are considered, the processes of general chemical analysis being fully and clearly described. In the fourth section the processes used for extracting, rendering, refining and bleaching oils, etc., are described, and here we have a most valuable compendium of information on a subject on which but little reliable and up-to-date information has hitherto been obtainable. In Section V. the author deals with the classification and uses of fixed oils, etc., and describes their adulterations and the methods of detecting them. Only some of the leading oils are considered in detail, but the information given is concise and reliable. The candle and soap industries are considered in the two concluding sections of the work. Here the author's special knowledge finds full scope.

Altogether, the book teems with information valuable alike to the analyst and the technical chemist. For many pages of the book the reader might suppose he was perusing a new edition of Mr. Allen's well-known volume II., while in other parts the author treats his subject from an entirely different standpoint, and introduces illustrations of manufacturing plant and processes which in themselves are sufficient to secure a warm welcome for the book.

The work is issued with cut leaves, is closely but clearly printed, and fairly free from typographical errors. It concludes with an index covering 45 pages.

APPOINTMENT.

Mr. George R. Thompson, Newport, Mon., has been appointed District Agricultural Analyst for the County of Monmouthshire and the County Borough of Newport.

THE ANALYST.

JULY, 1894.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on June 6th at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner took the chair.

The minutes of the last meeting were read and confirmed.

Mr. Allen then read a paper on "The Examination of Urine for Sugar," the publication of which is unavoidably held over.

Mr. Chattaway now read the two following papers :

ON THE COMPOSITION OF CHEESE.

BY WM. CHATTAWAY, T. H. PEARMAIN, AND C. G. MOOR.

WE were informed some months ago that a cheese was again being imported into this country in which the butter-fat had been largely replaced by other oils and fats.

We have not until recently succeeded in obtaining a specimen, but a sample of cheese, purchased under the Food and Drugs Act a short time ago, gave such abnormal results when examined by one of us, that we thought it would be of interest to compare in a table the figures representing the proximate constituents of the more commonly-used cheeses, which, so far as we are aware, have not recently been published.

We are unable to judge of the number of cheese samples examined last year under the Food and Drugs Act, but there is little cause for doubt but that the number is even more miserably disproportionate in the case of this commodity than it is in the case of many other articles.

In addition to this, we are aware that a cheese will shortly be placed on the market in this country in which the butter-fat is largely, if not entirely, replaced by other fats; and this cheese will doubtless be palmed off on the public as ordinary cheese, containing the due proportion of genuine butter-fat.

It would be out of place here to discuss the dietetic value of butter-fat over that of all other varieties; but it is insisted on by the best authorities that fat derived from milk is absorbed directly into the system, which is said to be the case of no other fat with which we are acquainted. Besides, it is clearly a fraud on the customer that any of the butter-fat should be abstracted, and an inferior, or, indeed, any other article substituted for it; for if the purchaser does not buy a cheese under a distinct title showing that it is made from skimmed or separated milk, he has a right to expect that the constituents in his cheese should bear the same proportions to one another as they do in ordinary milk.

It would be exceedingly interesting if it were possible to make any tests which would give an indication as to how far the fat and casein are assimilable, and which is most valuable from a food standpoint; but as we fear these have still to be discovered, we have endeavoured to make the chemical examination as complete as possible, and have to that end determined the following constituents, *i.e.*, water, ash, fat, and nitrogen, and have further examined the fat by the Reichert-Wollny and the Valenta test.

CHEESE RESULTS.

No. of Sample.	Name of Sample.	Water.	Ash.	Fat.	Reichert c.c. N	Nitrogen.	Casein.	Valenta Test.
1	Cheddar (English)...	33.0	4.3	29.5	24.	4.31	27.4	39.0° C.
2	Cheddar (English)...	35.5	4.2	25.6	28.8	4.39	27.8	42.0
3	Cheddar (English)...	33.8	4.1	30.5	26.4	4.20	26.7	31.0
4	Cheddar (Canadian)...	33.3	3.6	30.6	24.0	4.34	27.6	41.5
5	American ...	29.8	3.7	33.9	26.2	4.76	30.3	47.5
6	American ...	30.6	3.6	27.7	3.0	4.84	30.8	82.0
7	American ...	29.1	3.7	35.3	23.0	4.41	28.1	46.0
8	American ...	24.1	3.9	32.0	25.8	—	—	—
9	American ...	27.0	4.5	30.1	24.8	—	—	—
10	American ...	25.0	7.9	20.1	30.4	—	—	—
11	American ...	27.2	4.4	30.9	25.4	—	—	—
12	American ...	28.1	4.5	33.0	25.6	—	—	—
13	Gorgonzola ...	40.3	5.3	26.1	22.1	4.36	27.7	26.5
14	Gorgonzola ...	33.9	4.6	26.7	23.6	4.06	25.8	45.0
15	Dutch ...	41.8	6.3	10.6	27.0	5.11	32.5	40.0
16	Dutch ...	37.6	6.5	22.5	23.0	4.58	29.1	49.0
17	Gruyère ...	28.2	4.7	28.6	30.0	4.93	31.3	37.5
18	Gruyère ...	35.7	3.7	31.8	31.1	4.49	28.7	41.0
19	Stilton ...	19.4	2.6	42.2	29.0	4.73	21.1	38.5
20	Stilton ...	21.2	2.9	45.8	32.0	4.14	26.3	45.5
21	Cheshire ...	37.8	4.2	31.3	31.6	4.03	25.7	43.0
22	Cheshire ...	31.6	4.4	35.3	31.8	4.16	26.5	47.0
23	Double Gloucester...	33.1	5.0	23.5	31.4	4.99	31.8	38.0
24	Double Gloucester...	37.4	4.6	28.1	32.3	4.45	28.3	41.0
25	Camembert...	47.9	4.7	41.9	31.0	3.43	21.8	32.0
26	Camembert...	43.4	3.8	22.6	35.0	3.83	24.4	33.0
27	Parmesan ...	32.5	6.2	17.1	28.0	6.86	43.6	28.0
28	Roquefort ...	29.6	6.7	30.3	36.8	4.45	28.3	19.0
29	Double Cream ...	57.6	3.4	39.3	31.2	3.14	19.0	40.0
30	Bondon ...	39.5	0.7	24.4	29.1	1.48	9.4	42.0
31	Cream (York)	63.1	1.4	6.5	29.0	2.76	17.9	41.0

The following figures call for notice, *viz.*, water in the Camemberts, Nos. 25 and 26, 48 and 43 per cent. respectively; the York cream 63 per cent., and the double cream 57.

Fat is very deficient in the following: York cream, 6.5; Parmesan, 17.1; and Dutch, No. 1, 10.6.

The nitrogen is very low in the Bondon cream, namely, 1.48; in the York cream it is 2.6, and in the Parmesan reaches the highest amount of 6.86 per cent.

From the above figures we should conclude that none of the cheeses (excepting the Dutch and the so-called "cream" cheeses) have been prepared from milk from which fat has been removed. The Parmesan cheese is, however, low in fat.

We wish to call particular attention to the American cheese, No. 6. This is a margarine cheese.

We read in Battershall's "Food Adulteration" that in 1885 a special New York State Brand was adopted for "*pure cream cheese*," which he says had a very good effect, and accomplished much in the restriction of the sale of the spurious article.

In certain previous papers on the subject of cheese, it is stated that various anomalies may be introduced by the production of varying quantities of free fatty acids, ammonia, and lactic acid, as the ripening process goes on, and, with a view of ascertaining what changes take place, we intend to examine from time to time samples, which we are preserving, of the more commonly-used cheeses, particularly from a bacteriological aspect; and we hope to lay the results before the Society at no distant date.

As to the cause of the changes in composition, taste, and smell, we are satisfied that they are almost entirely due, in the first instance, to organized ferments, which may be checked or encouraged at will according to the conditions under which we cause the ripening to take place.

Seeing that digestible nitrogenous matter is thus converted into ammonia compounds, it would appear that, as far as the food value is concerned, the further the ripening or decomposition is carried the greater the loss of flesh-forming constituents.

We have in no case found boric acid present in any of the cheeses.

We have not examined the colouring matter either in the cheeses themselves or in their rind.

In conclusion, we would urge that cheese should not be so neglected by analysts and inspectors, and that attention should be more particularly directed to American cheeses.

Our thanks are due to Mr. Robert Waterhouse and Mr. Martin Priest for their valuable assistance in connection with this paper.

NOTE ON THE VALENTA ACETIC ACID TEST.

By WM. CHATTAWAY, T. H. PEARMAIN AND C. G. MOOR.

VALENTA'S test has given in our hands such general satisfactory results that we are somewhat surprised that it has not held a more important position than it appears to have done. The acetic acid turbidity temperature has the undeniable disadvantage of being one of those empirical methods of analysis which cannot be regarded with the same satisfaction as the many estimations which are so definite as to be expressed in the form of a chemical equation. We contend, however, that notwithstanding this fact, the value of this test is by no means small, and, further, that this disadvantage equally applies to the Reichert-Wollny, the Maumène and other important determinations.

Certain precautions have to be noted in order to obtain concordant observations, and we wish to call attention to the following points :

- (1) Strength of acetic acid.
- (2) Presence of water in the oil or fat under examination.
- (3) The temperature to which the oil or fat has been previously heated.
- (4) The method of stirring and observing the turbidity point.

1. We have paid considerable attention to the most suitable strength of acid to use, and after a somewhat lengthy trial have adopted an acid which contains 99.50 per cent of acetic acid.

In this connection we would like to call attention to the remarks made by Mr A. H. Allen in his "Commercial Organic Analysis," vol. ii., p. 26, in which he says that a slight variation in the strength of the acid employed is not of great importance. This is not in accordance with our experience, when working with an acid of the above-stated strength, and we believe that as the amount of water is increased the extreme sensitiveness of the turbidity test is diminished. On the other hand, there are certain reasons why an absolute acetic acid should not be used. It will be seen from this that acid of the strength specified by Valenta is more liable to give a little latitude in the exact strength of the acid to be employed, but that while such is the case, this advantage is entirely outweighed from the standpoint of sensitiveness.

2. The presence of moisture in the fat or oil under examination is probably one of the most fruitful sources of error, and we have found that in all cases it is advisable to filter the samples through a perfectly dry filter paper before making the test. It is well known to oil importers that the presence of minute quantities of water in the oil seriously affects the appearance of the oil as soon as the temperature falls, and that an oil may be quite brilliant and yet contain an estimable quantity of water. We believe that this fact was not quite recognised by Valenta, and also that it explains, or partly explains, the difference in figures obtained by Valenta in the same class of oil.

Olive-oil has shown the most serious variations in this respect, and it is the oil most likely to contain traces of water, owing to the methods of pressing and the variable condition of the fruit at the time it was pressed.

3. When an oil or fat is unduly heated, either at the time the test is being made or previously, the determination of the turbidity figure must not be relied upon. In all cases the sample must be dealt with as cautiously as it can be in this respect, and we would recommend that the temperature of 100°C. should never be exceeded in the preparation of an oil or fat for the test.

4. The method which is usually adopted consists in stirring together by means of a thermometer equal volumes of acetic acid and the sample in a test-tube after the solution is effected, removing the source of heat, and noting the temperature at which turbidity appears.

This is very well as a rough sorting test, and answers well enough in many cases; but we have now adopted the plan of conducting the test in a bath of water which has been raised to a suitable temperature; by these means we obviate an error which arises from the unequal cooling of the test-tube, and instead of a kind of tiny

waterspout appearing in the tube, the whole of the contents of the tube become turbid instead of doing so locally.

We would like to call attention to a comparatively perfect method of working the test, which, notwithstanding its extreme simplicity, is capable of giving results which may be regarded as absolutely concordant.

In some methods of analysis it is not expedient to arrive at very exact results owing to length of time necessary, but we hope it will be generally regarded by this society that a simple test like this, which enables a reliable, and even quantitative, result to be obtained in a few minutes, will allow of a little time being spent in order to obtain highly concordant figures.

Instead of using an ordinary test-tube, we use a short and somewhat thick test-tube of about $4" \times \frac{1}{2}"$, into which a well-fitting stopper has been ground. We then weigh into the tube 2.75 grammes of the fat, and measure from a burette, or other suitable arrangement, 3.0 c.c. of the acetic acid. The tube is then stoppered, and placed in a beaker of warm water, increasing the heat until, after well shaking the tube, the contents become quite clear. The source of heat is then removed, and the test-tube so placed that it is in the centre of the beaker of heated water, and by means of a thermometer attached to the test-tube by a rubber band, the whole is allowed to rest until we note the change from brilliancy to turbidity. The change is very definite, and can be repeated over and over again with a maximum error of about 0.25 of a degree. If preferred, the thermometer may be used as a thermometer stopper to the test-tube, but it is not in our opinion necessary, as it is not always convenient to have a few dozens of thermometer-stoppered tubes, whereas simply stoppered tubes are more easily obtained and equally satisfactory.

It occurs to us that the foregoing remarks may appear to reflect to some extent upon the value of the Reichert-Wollny and other determinations. Such is very far from our intention. Food analysis is now no simple matter, and every care has to be exercised before one is in a position to state whether a food sample is genuine or not.

We realize that every determination that can throw light, however small, upon the analysis of food, and particularly of such commodities as butter and cheese, is most valuable. In this sense, and in this purely, we would suggest that the use of this method of analysis be taken up by those who have not already done so.

Finally, we do not wish to place the Valenta test in too high a position. There are still some oils which give strange and apparently inconsistent results.

The investigations into these irregularities are already under our consideration, and we believe that they are capable of simple explanation in most cases. It must not, however, be supposed that an oil may be purified by several methods, and that then each of such purified samples will give the same result. We repeat that this is a sensitive test, and as such it is quite inconsistent to expect that under such conditions as those cited we should not get a different result.

Its use as a valuable indication as to the nature of butter, margarine proper, and mixtures of these is now established.

We have placed on the blackboard just one instance of this—a sample of margarine, and of butter, and definite mixtures of the same.

The calculations are based upon the figures obtained from the butter and margarine proper as initial figures; but these figures are quite within the usual range, and these determinations were made without any important care, such as that specified in the improved method to which we have referred:

PRESENT.			FOUND.	
Mixture.	Butter.	Margarine.	Butter.	Margarine.
1	10	90	8.6	91.4
2	20	80	20.7	79.3
3	30	70	31.0	69.0
4	40	60	41.3	58.7
5	50	50	50.0	50.0
6	60	40	58.6	41.4
7	70	30	70.7	29.3
8	80	20	81.0	19.0
9	90	10	93.1	6.9

The following table contains some further figures taken upon a variety of pure oils, which we think may be of some interest. These results, however, must not be regarded as standards:

ACETIC ACID TURBIDITY TEST.

	°C.		°C.		°C.
Butter-fat:		Cottonseed ...	75.0	Japan Fish	19.0
(Number of samples examined, 24)		" ...	71.0	Herring ...	90.0
Highest ...	39.0	" ...	85.0	Nigerseed ...	68.5
Lowest ...	29.0	" ...	86.0	Sunflower ...	59.0
Mean ...	36.0	" ...	89.0	" ...	62.5
Margarine:		" ...	88.0	Bottlenose ...	80.0
(Number of samples examined, 5)		Cod ...	31.0	" ...	96.0
Highest ...	97.0	" ...	29.0	Lard-oil ...	75.0
Lowest ...	94.0	" ...	26.5	" ...	76.0
Mean ...	95.5	liver ...	76.0	" ...	75.0
Olive (Bari) ...	91.0	" ...	72.0	Lard ...	98.0
" (Smyrna) ...	91.0	" ...	73.0	" ...	97.0
" (Lucca) ...	90.0	Colza (German) ...	83.0	" ...	98.0
" ...	91.0	Rape ...	63.0	" ...	97.0
" ...	89.0	" ...	77.0	Neat's-foot ...	72.0
" ...	86.0	" ...	77.0	Tea ...	78.0
" ...	85.0	" ...	78.0	Rosin ...	56.0
" ...	90.0	Peach-kernel ...	82.0	" ...	42.0
" ...	83.0	" ...	82.0	Jamba ...	above 100.0
" ...	85.0	Ground-nut ...	72.0	" ...	100.0
Almond ...	87.0	" ...	73.0	" ...	100.0
" ...	84.0	" (Arachis) ...	73.5	Cabbage ...	100.0
" ...	82.0	Linseed ...	46.0	Beef-stearin ...	100.0
" ...	82.0	" ...	52.0	Lard ...	100.0
" ...	72.0	Seal ...	48.0	Castor not above	18.0
Cottonseed ...	71.0	" ...	70.0	Woolgrease olein	18.0
		Japan Fish ...	47.5	not above	18.0

In the absence of Mr. Jones, Mr. Bevan read the following paper :

TURBIDITY TEMPERATURE OF OILS AND FATS WITH GLACIAL ACETIC ACID.

By E. W. T. JONES.

THIS test, due to Valenta, is described by him in J. C. S., xlvii, p. 1078, where he also gives results on certain oils.

I have been investigating this test, and although the hopes I had entertained of its usefulness, as an independent and totally distinct test for differentiating between those butters which, although genuine, are low in the usual butter characteristics according to chemical testing, and actual mixtures approximating such butters, have not been realized, I have ascertained a few facts concerning the test which I think are worth recording.

Valenta indicates the requisite strength of the acid by specific gravity, which he gives as 1056.2. This, according to the most reliable table I can find (Oudemans'), may mean 44 per cent. $\text{H}\bar{\text{A}}$, or 99.7 per cent. $\text{H}\bar{\text{A}}$. Of course the former strength is not "glacial," but I shall show that specific gravity is altogether inadequate to determine the fitness of the acid for oil-testing; indeed, it will need no further demonstration when I mention that 4 c.c. of water, added to 2,300 c.c. (a Winchester quart) of acid, lowered the turbidity temperature 5°C . This means that a difference of less than 0.2 per cent. in the $\text{H}\bar{\text{A}}$ effects this divergence, and it follows that a very small dilution of the strongest acid may make it absolutely useless for the test at all. The turbidity temperature, say, with a known sample of butter-fat, is the most delicate test I know of for adjusting the requisite strength of this strong acetic acid for the purpose in view.

An acid which by titration contained 98 per cent. (97.99) of $\text{H}\bar{\text{A}}$ gave with a butter-fat a temp. of 48°C .; the addition of 2 per cent. of water made it over 100°C .—quite unfit for use; 1 per cent. made it 79°C ., whilst 0.2 per cent. made it 55°C . This shows the importance, if this test is to be used to give at all comparable results, of adopting a scheme to secure exactly the right strength of acid, and the following is the course I have followed.

I have carefully filtered off some fat from a normal butter into a bottle, and this I employ to set every fresh batch of acid I use. I procure the best and strongest glacial acetic acid, and suppose I find it gives a temp. of 50°C . with my standard butter-fat, I carefully add water until the temp. with it is 60°C .; thus I know that whatever tests I make with this acid are strictly comparable with any previous tests I have made.

It is most important, too, that the relative proportions of the acid and fat or oil are pretty strictly adhered to, and absolutely necessary that the set proportion of the fat or oil to the acid is not exceeded; any error in measurement should lean to the full measurement of the acid, which does not make the difference that the converse proportion does. I make special pipettes, a narrow one delivering 3 grammes of water at 15.5°C . for the acid, and wider ones, with wider orifices, delivering

3 grammes of water at 15.5° C. for the fat. I measure all oils and fats at about 50° C.

The turbidity temperature of butter-fat varies from 40° to 70° C.—the general run being from 52° to 65° C. The following analyses show the temp. in conjunction with other estimations.

Turbidity temperature.	54° C.	56° C.	55° C.	60° C.	55° C.	56° C.	57° C.	41° C.	45° C.	59° C.	48° C.
Koettstorfer's deg.	226.8	224.0	226.2	220.0	223.4	226.2	225.1	224.5	225.7	217.8	221.2
Soluble acids ...	5.14	4.79	5.19	4.19	5.02	5.30	5.20	4.97	5.05	4.12	4.98
Insoluble acids ...	88.92	89.30	89.04	90.28	89.08	88.78	89.11	88.74	88.56	90.18	88.73
Reichert-Wollny ...	27.2	25.6	28.1	22.5	26.7	28.2	28.1	26.7	27.2	22.2	25.4
Equal to volatile acid, as butyric ...	4.79	4.50	4.95	3.96	4.69	4.97	4.95	4.69	4.79	3.91	4.47

Another sample had a temp. of 71° C., and gave a Reichert figure 23.9.

Margarine gives temp. 95° to 106° C., generally from 100° to 102° C.

A mixture of 4 parts of butter-fat, having temp. of 40° C., and 1 part margarine, having temp. of 95° C. gave a temp. of 52° C., against 51 calculated.

Another mixture of 27 parts butter-fat, of temp. 52° C., and 73 parts margarine, of temp. 105° C., gave a temp. of 92° C., against 90.7° C. calculated.

The following are the turbidity temperatures of other oils and fats I have tried with this same acetic acid :

Rape oil	101° C.
Sesame oil	77°
Linseed oil (i.)	53°
Linseed oil (ii.)	57°
Lard oil	96°
Cottonseed oil	76°
Olive oil	89°
Arachis oil (i.)	61°
Arachis oil (ii.)	88°

If any member desires to adopt the same strength of acid as used in my experiments, I shall be happy to communicate with him for the purpose.

DISCUSSION.

Mr. Allen thought the society were very much indebted to the authors of the paper on the acetic acid test, for the observations they had brought forward, and for their improved method of operating. He did not quite know why the authors adopted the weight of 2.75 grammes of oil instead of taking a measure of 3 c.c. It was adding a complication to the test, which he thought was not capable of giving results other than what might be called of a preliminary kind. He had met with acetic acid which it was necessary to freeze in order to render it strong enough to use for the test; but he had never realized that it was necessary to employ such very strong acid as the authors preferred. In stating as he did in 1866 in "Commercial

Organic Analysis" that the strength of the acetic acid employed was not of great importance, he had in mind the comparative results yielded by different oils, rather than the absolute turbidity temperatures. There was no doubt that the weaker the acid was the higher became the turbidity-point with all butters; but, then, the margarine point rose also. As Mr. Jones had stated, it was necessary to have a standard sample of butter with which to compare others. When the Valenta test for oils was first published, he suggested its possible application to butter, and it had since been used in his laboratory on several thousands of samples. As a preliminary or sorting test, he had found it exceedingly useful. The authors' experience showed that genuine butters gave turbidity temperatures varying as much as 10° C., so that the quantitative results could not be very precise; but the same objection applied to every test for butter, and as this was based on an entirely different principle from most others, it was distinctly of value; and he was glad to see that it had assumed an improved form. He was also interested to see that the olive oils gave such approximately constant results. He observed that figures for three specimens of cod oil and three specimens of cod-liver oil were given in the table. He would like to know whether Mr. Chattaway knew the history of these cod oils. When it came to the point, "cod oil" meant, or should mean, cod-liver oil. Hence, it was clear that, if the oil really came from the cod, there was no necessity to insert the word "liver," and therefore the cause of the difference in the results obtained was not apparent. The influence of the volume of acetic acid used had been investigated by Mr. G. H. Hurst (*Jour. Soc. Chem. Ind.*, vi. 22), who had also tried the plan of surrounding the tube with a beaker of water.

Mr. H. Droop Richmond wished to know how the strength of the acetic acid was determined. Titration was not a very accurate method when the acid was nearly 100 per cent. The indicator to be used had to be considered; and it was also possible to over-estimate, according to the carbon dioxide present in the solutions of the acid. It was further possible to under-estimate, according to the amount of carbon dioxide in the standard solution against which the alkaline solution was standardized, and these two errors would not necessarily compensate each other. He believed that the density would be the best means of estimating the strength, but it was not actually known what the density of 100 per cent. acetic acid was. Some tables had been published, but they did not agree very well. By far the best table was that of Oudemans, but it only extended to four places of decimals, and one could not be certain whether it was accurate to the fourth place. He had made some experiments which seemed to him to indicate that water was soluble in butter-fat to the extent of nearly a quarter of a per cent. He did not agree with Mr. Allen in thinking that Valenta's test was only of value as a preliminary one. He believed that it was almost as good a test as the density, and, in combination with others, promised to be valuable. As for the Reichert-Wollny, he thought it should not be too implicitly relied upon, as many methods which were looked upon as preliminary showed actually less variation in genuine samples. With regard to cheese, there were two rather interesting determinations, showing that the butter of sheep's milk differed from that of the cow, though Besana had not found any marked difference (Nos. 26 and 28 in the Reichert-Wollny figures). Sample No. 27, Parmesan cheese, was prepared from

skimmed milk. He thought that, as a general rule, all samples were distinctly suspicious in which the fat fell below the casein, estimated, as the authors of the paper had estimated it, by determining the nitrogen and multiplying by the factor, 6.38. Had Mr. Chattaway determined the amount of sodium chloride in the ash?

Mr. R. Bodmer found that the great trouble of the test was that no acetic acid of the requisite strength was readily obtainable.

The Chairman (Mr. Hehner) said that the figures did not show how nearly margarine could be determined in commercial samples, but how accurately two substances, separately known, could be re-estimated after having been mixed. He had experimented a good deal with the Valenta test, and once thought highly of it; but on fuller experience he had been disappointed, as was the case with all other methods of fat-analysis. Figures obtained from the examination of a limited number of samples always broke down on wider experience. Further, a slight alteration in the strength of the acid made a considerable difference in the figures. Even when using acetic acid from the same stock-bottle, the latter having been opened frequently, the acid changed in its action when the bottle was getting empty. The state of freshness of the butter also required consideration. In the acetic turbidity table given by the authors there were shown the results of 24 samples of butter-fat, and in a second table the Valenta figures of 26 samples of cheese-fat. The fresh butter-fats gave figures which varied only by 10° C.; but in the fats obtained from those cheeses which according to the Reichert number were almost certainly genuine and free from margarine, and carefully excluding cheeses which appeared to be "filled," the variation was from 19° to 45° C. Even excluding the Roquefort cheese, the variation was yet so great as to allow of the introduction of a large percentage of margarine, that percentage being not less than that covered by the variations in the figures obtained by other processes. That was the conclusion which was forced upon him by his practice in the laboratory, and which he was confirmed in, subject to any explanation which the writers of the paper now under discussion might offer. A very interesting point was raised which had been frequently before his mind; that was, What was the real significance of the Valenta test? Mr. Richmond justly endeavoured to attain the highest possible accuracy in everything. If the significance of the figures was not known, it seemed to him this high degree of accuracy was largely wasted. He had made inquiry into this matter, and found that the fat which crystallized from the acetic acid consisted of the saturated fatty compounds, whilst the unsaturated remained in solution. It seemed to him that these small mixtures up to about 15 or 20 per cent. were as undetectable by the Valenta as by any other process.

Mr. Cassal would like to know whether, in the authors' experience, the Valenta test was one which could be relied upon for the quantitative analysis of samples of butter, as the other processes now used could be relied upon. At first sight the authors' table appeared to be remarkable, and it looked as if they had arrived, by the use of the Valenta test, at a process whereby the amount of margarine that was actually present in an adulterated butter could be determined with an extraordinary degree of accuracy. But he understood that this table was merely intended

to represent the powers of the Valenta test when applied to mixtures of a particular butter and a particular margarine separately giving certain results with the Valenta test. There was, however, nothing to show that upon taking any mixed sample the Valenta test applied to it would enable one to detect the percentage of the adulterant with anything like such accuracy. Great caution should be exercised in drawing conclusions from a test founded upon conditions as yet not fully determined, and in regard to which but little in the way of satisfactory scientific explanation had as yet been given.

Mr. Chattaway, in reply, said that the reason why 2.57 grammes of fat were taken was with a view to as nearly as possible approximate the weight of 3 c.c. at a temperature of 40° C. He did not approve of Mr. Allen denoting the method as a mere sorting test. It was, in the opinion of the authors, quite as useful and valuable, if not more so, than the Reichert-Wollny. He could have wished not to have occasion to speak so strongly in favour of the Valenta test, seeing that it is not as yet fully understood; but the authors had every reason to regard the test as decidedly more valuable than the Reichert determination. The butters mentioned were taken from some examined during the past few weeks, and although not truly representing the *possible* variations of butter, they were fairly representative of the butters of commerce. The same remarks applied also to the margarines, the figures of which are recorded. So far as the table to which Mr. Cassal and Mr. Allen had called attention was concerned, the authors wished particularly that it should not be misunderstood. A butter and margarine were taken, mixtures made, and then, starting with the initial figures of the butter and margarine, the calculations were made. The cod oil was a crude specimen used for leather dressing. The authors had been able to buy the acid quite strong enough; indeed, they had had to dilute it down to the proper strength. Referring to Mr. Richmond's observations, the strength of the acid was determined as usual by baryta and phenolphthalein, and, in the opinion of the authors, this was quite satisfactory. Mr. Chattaway thought that the attempt to place the test in a higher position than it had usually held had been severely—too severely—dealt with by Mr. Hehner. It seemed that Mr. Hehner did not so much really disapprove of the test, as that he did not think *any* test was at the present moment perfect enough to deal with certain cases. Touching again the question of sensitiveness of the test, the authors were in a position to prove that, taking hundreds of figures and determinations into consideration, the Valenta test was capable of just twice the accuracy of the Reichert-Wollny. The explanation of the solution at certain temperatures of some fats and not others was dependent, in the speaker's belief, on the intermixibility question. Butyric acid, for example, existing in butter in combination with glycerin was readily soluble in acetic acid at a certain temperature, and this mixture then had the power of taking into solution the glycerin compounds of the higher fatty acids. The authors did not wish to place too much stress upon the Valenta figures obtained in the case of fat from cheese, owing to the imperfect knowledge of the changes which take place in the maturing of cheese.

Members of the Society are requested to send any remarks or suggestions they may desire to make in reference to the following Draft to the Secretaries, without delay, and in as condensed and systematic a manner as possible. The Committee of the House of Commons is now sitting and receiving evidence on the working of the Food Acts.

DRAFT OF A SALE OF FOOD AND DRUGS ACT, PREPARED BY THE COUNCIL OF THE SOCIETY OF PUBLIC ANALYSTS,

JUNE, 1894.

AN ACT TO AMEND THE ADULTERATION OF FOOD ACTS, AND TO MAKE BETTER PROVISION FOR THE SALE OF FOOD AND DRUGS IN A PURE STATE.

WHEREAS it is desirable that the Acts now in force relating to the adulteration of food should be repealed, and that the law regarding the sale of food and drugs in a pure and genuine condition should be amended :

Be it therefore enacted by the Queen's most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal and Commons in this present Parliament assembled, and by the authority of the same, as follows :

1. From the commencement of this Act the statutes of the twenty-third and twenty-fourth of Victoria, chapter eighty-four of the thirty-first and thirty-second of Victoria, chapter one hundred and twenty-one, section twenty-four, of the thirty-third and thirty-fourth of Victoria, chapter twenty-six, section three, and of the thirty-fifth and thirty-sixth of Victoria, chapter seventy-four of the thirty-eighth and thirty-ninth of Victoria, chapter sixty-three, and of the forty-second and forty-third of Victoria, chapter thirty, shall be repealed, except in regard to any appointment made under them and not then determined, and in regard to any offence committed against them or any prosecution or other act commenced and not concluded or completed, and any payment of money then due in respect of any provision thereof.

2. The term "food" shall include every article used for food or drink by man, other than drugs or water, or any article intended to enter into or be used in the preparation of human food, and all flavouring matters and condiments.

The term "drug" shall include medicines for external or internal use.

The term "county" shall include every county, riding, and division, as well as every county of a city or town not being a borough.

The term "justices" shall include any police and stipendiary magistrate invested with the powers of a justice of the peace in England, and any divisional justices in Ireland.

DESCRIPTION OF OFFENCES.

3. No person shall mix, colour, stain, or powder, or order or permit any other person to mix, colour, stain, or powder, any article of food with any ingredient or material so as to render the article injurious to health, with intent that the same

may be sold in that state, and no person shall sell any such article so mixed, coloured, stained, or powdered, under a penalty in each case not exceeding fifty pounds for the first offence; every offence, after a conviction for a first offence, shall be a misdemeanour, for which the person, on conviction, shall be imprisoned for a period not exceeding six months with hard labour.

4. No person shall, except for the purpose of compounding as hereinafter described, mix, colour, stain, or powder, or order or permit any other person to mix, colour, stain, or powder, any drug with any ingredient or material so as to affect injuriously the quality or potency of such drug, with intent that the same may be sold in that state, and no person shall sell any such drug so mixed, coloured, stained, or powdered, under the same penalty in each case respectively as in the preceding section for a first and subsequent offence.

5. Provided that no person shall be liable to be convicted under either of the two last foregoing sections of this Act in respect of the sale of any article of food, or of any drug, if he shows to the satisfaction of the justice or court before whom he is charged that he did not know of the article of food or drug sold by him being so mixed, coloured, stained, or powdered as in either of those sections mentioned, and that he could not with reasonable diligence have obtained that knowledge.

6. No person shall sell to the prejudice of the purchaser any article of food or any drug which is not of the nature, substance, and quality of the article demanded by such purchaser, under a penalty not exceeding twenty pounds.

In any prosecution under this Act for selling to the prejudice of the purchaser any article of food or any drug which is not of the nature, substance, and quality of the article demanded by such purchaser, it shall be no defence to any such prosecution to allege that the purchaser, having bought only for analysis, was not prejudiced by such sale. Neither shall it be a good defence to prove that the article of food or drug in question, though defective in nature or in substance or in quality, was not defective in all three respects.

Any drug sold under any name included in the British Pharmacopœia shall be required to comply with the description, character, and tests for such drug as specified in the latest edition, with amendments, of the "British Pharmacopœia," provided that the drug be not included in the list of exceptions included in Schedule II. of this Act.

7. No person shall sell any compound article of food or compounded drug which is not composed of ingredients in accordance with the demand of the purchaser, under a penalty not exceeding twenty pounds.

8. Provided that no person shall be guilty of any such offence as aforesaid in respect of the sale of an article of food or a drug not recognised by the British Pharmacopœia mixed with any matter or ingredient not injurious to health, and not intended fraudulently to increase its bulk, weight, or measure, or to conceal its inferior quality, or any article of food or a drug deficient in any material constituent, if at the time of delivering such article of food or drug he shall supply to the person receiving the same a notice, by a label distinctly and legibly written or

printed, affixed to the containing vessel or parcel, and on the outside wrappers of the article or drug, in characters more prominent than any others, upon any label or other thing upon or with such article of food or drug, describing such article of food or drug as a mixture, and naming the ingredients thereof and their relative proportions, or declaring its deficiency or deficiencies.

It shall be no defence, under this section, to plead the accidental deterioration of the article, or accidental abstraction or spontaneous separation or evaporation of the ingredients.

APPOINTMENT AND DUTIES OF ANALYSTS, AND PROCEEDINGS TO OBTAIN ANALYSIS.

10. In the city of London and the liberties thereof the Commissioners of Sewers of the city of London and the liberties thereof, and in all other parts of the Metropolis, the vestries and district boards acting in execution of the Act for the better local management of the metropolis, the court of quarter sessions of every county, and the town council of every borough having a separate court of quarter sessions, or having under any general or local Act of Parliament or otherwise a separate police establishment, may, as soon as convenient after the passing of this Act, where no appointment has been hitherto made, and in all cases as and when vacancies in the office occur, or when required so to do by the Local Government Board, shall for their respective city, districts, counties, or boroughs, appoint one or more persons possessing competent knowledge, skill and experience, as analysts of all articles of food and drugs sold within the said city, Metropolitan districts, counties, or boroughs, and shall pay to such analysts such remuneration as shall be mutually agreed upon, and they shall not remove them during good behaviour, and all appointments, remunerations, and removals shall at all times be subject to the approval of the Local Government Board, in consultation with the Board of Reference provided for in section 30, who may require satisfactory proof of competency to be supplied to them, and give their approval absolutely, or with such modifications as to the conditions of appointment as they may deem desirable. Provided, that no person shall hereafter be appointed an analyst for any place under this section who shall be engaged directly or indirectly in the manufacture or sale of food or drugs in such place.

In Scotland the like powers shall be conferred and the like duties shall be imposed upon the commissioners of supply at their ordinary meetings for counties, and the commissioners or boards of police, or where there are no such commissioners or boards, upon the town councils for boroughs within their several jurisdictions; provided that one of Her Majesty's Principal Secretaries of State in Scotland shall be substituted for the Local Government Board of England.

In Ireland the like powers and duties shall be conferred and imposed respectively upon the grand jury of every county and town council of every borough; provided that the Local Government Board of Ireland shall be substituted for the Local Government Board of England.

12. Any purchaser of an article of food or of a drug in any place being a district, county, city, or borough where there is any analyst appointed under this

or any Act hereby repealed shall be entitled, on payment to such analyst of ten shillings and sixpence, or if there be no such analyst then acting for such place, to the analyst of another place of such sum as may be agreed upon between such person and the analyst, to have such article analysed by such analyst, and to receive from him a certificate of the result of his analysis.

13. (a) Any medical officer of health, inspector of nuisances, or inspector of weights and measures, or any inspector of a market, or any police constable, or other duly authorized person under the direction and at the cost of the local authority appointing such officer, inspector, or constable, or charged with the execution of this Act, may procure any sample of food or drugs, and shall submit the same to be analysed by the analyst of the district or place for which he acts, or if there be no such analyst then acting for such place to the analyst of another place, and such analyst shall, with all convenient speed, analyse the same and give a certificate to such officer, wherein he shall specify the result of the analysis.

(b) Any person authorized to take samples under this clause may procure at any railway-station, or elsewhere during transit, whether within or outside of the district in which he is empowered to act, or at the place of delivery, any sample of any article of food or drug which may be consigned or addressed to any person or persons within the limits of his district in pursuance of any contract for the sale to such consignee, and such officer shall submit the same to the public analyst of the district appointing him, and the same shall be analysed, and proceedings shall be taken and penalties, on conviction, be enforced in the like manner in all respects as if such officer, inspector, or other person had purchased the same from the seller or consignor under section 13 (a) of this Act.

The seller or consignor or any person or persons entrusted by him for the time being with the charge of such article, if he shall refuse to allow such officer, inspector, or constable to take the quantity which such officer, inspector, or constable shall require for the purpose of an analysis, shall be liable to a fine not exceeding ten pounds.

14. The person purchasing any article with the intention of submitting the same to analysis shall, after the purchase shall have been completed, forthwith notify to the seller, or his agent selling the article, his intention to have the same analysed by the public analyst, and shall, except in cases covered by clause 15, offer to divide the article into three parts to be then and there separated, and each part to be marked and sealed or fastened up in such manner as its nature will permit, and shall, if required to do so, proceed accordingly, and shall leave one of the parts with, or cause it to be delivered to, the seller or his agent.

He shall afterwards retain one of the said parts for future comparison, excepting in cases covered by clause 15 of this Act, and submit the third part to the analyst.

15. If the seller, or his agent, do not accept the offer of the purchaser to divide the article purchased in his presence, the analyst receiving the article for analysis shall divide the same into two parts, and shall seal or fasten up one of those parts and shall cause it to be delivered, either upon receipt of the sample or when he

supplies his certificate to the purchaser, who shall retain the same for production in case proceedings shall afterwards be taken in the matter.

In cases where the article purchased cannot be conveniently divided or where its division at the time of sale would militate against the accuracy of the analysis or where samples are taken under section 13 (a), the whole of the article purchased shall be submitted to the analyst, who shall divide the same into two parts and fasten up and seal one of those parts, and shall cause it to be delivered, either upon receipt of the sample, or when he supplies his certificate, to the purchaser, who shall retain the same for production in court in case proceedings be taken. If the course of procedure directed by clause 14 has been omitted owing to the nature of the article, and not by refusal of the vendor to have the division forthwith made, the analyst shall specially report in his certificate whether the article was of such a character as to justify a departure from the course directed in clause 14.

16. A person requiring an article to be analysed may deliver the same personally, or by deputy, at the office of the analyst, or such article may be forwarded to the analyst by registered post, and the charge for the transmission of such article shall be deemed one of the charges under this Act or of the prosecution, as the case may be, and samples so forwarded shall be deemed to be delivered by the purchaser.

17. If any such officer, inspector, or constable, as above described, shall apply to purchase any article of food or any drug exposed to sale, or intended for sale on any premises or in any shop or stores, and shall tender the price for the quantity which he shall require for the purpose of analysis, not being more than shall be reasonably requisite, and the person exposing the same for sale shall refuse to sell the same to such officer, inspector, or constable, such person shall be liable to a penalty not exceeding ten pounds.

Any officer, inspector, or constable, as above described, may, without going through the form of purchase, but otherwise acting in all respects with the aforesaid provisions of the Act, take from any shop, premises, store, warehouse, or in any street or open place of public resort, such quantity of any article of food and drug exposed for sale, on sale, or intended for sale, as may be required for the purpose of analysis, tendering the price for the quantity he shall require. Any dealer or his agent who shall refuse to supply the said article to such officer, inspector, or constable, shall be liable to a penalty not exceeding ten pounds. Any street or open place of public resort shall be held to come within the meaning of this section.

18. The certificate of the analysis shall be in the form set forth in the schedule hereto, or the like effect, and shall, when reasonably practicable, state the nature and proportion or proportions of such foreign ingredient or ingredients found on analysis, deficiency of strength or extent of deterioration or evaporation, but the analyst shall not be required to give the analytical data in his certificate on which his opinion is based.

18a. Every Local Authority empowered under sections 10 and 11 of this Act shall cause one or more of the officers specified under clause 13 of this Act to submit samples to the public analyst for analysis, the number of such samples being not less

than one sample per annum for every 1,000 inhabitants living within the area of such Local Authority.

Should such Local Authority refuse or neglect to submit samples for analysis, or submit a smaller number than one for every 1,000 inhabitants per annum, the Local Government Board may appoint an officer or officers for the purpose of obtaining samples for analysis by the public analyst, and to take proceedings against offenders. The cost of such appointment, purchase, and proceedings shall be borne by the Local Authority or Authorities.

19. Every analyst appointed under any Act hereby repealed or this Act shall report quarterly to the Authority appointing him the number of articles analysed by him under this Act during the foregoing quarter, and shall specify the result of each analysis and the sum paid to him in respect thereof, and such report shall be presented at the next meeting of the Authority appointing such analyst, and every such Authority shall annually transmit to the Local Government Board, at such time and in such form as the Board shall direct, a certified copy of each such quarterly report.

PROCEEDINGS AGAINST OFFENDERS.

20. When the analyst, having analysed any article, shall have given his certificate of the result, from which it may appear that an offence, or offences, against one or more of the provisions of this Act has been committed, the person causing the analysis to be made may take proceedings for the recovery of the penalty, or penalties, herein imposed for such offence, or offences, before any justices in petty sessions assembled having jurisdiction in the place where the article sold was delivered to, or obtained by, him in a summary manner.

In all prosecutions under this Act, except as hereinafter provided, the summons to appear before the magistrates shall be served upon the person or persons charged with violating the provisions of the said Act within a reasonable time, and in the case of a perishable article, not exceeding twenty-eight days from the time of the purchase from such person of the food or drug, for the sale of which in contravention to the terms of the Act the vendor is rendered liable to prosecution, and particulars of the offence or offences against the said Act of which the seller is accused, and also the name of the prosecutor, shall be stated on the summons, and the summons shall not be made returnable in a less time than seven days from the day it is served upon the person or persons summoned.

Every penalty imposed by this Act shall be recovered in England in the manner prescribed by the eleventh and twelfth Victoria, chapter forty-three. In Ireland such penalties and proceedings shall be recoverable, and may be taken with respect to the police district of Dublin metropolis, subject and according to the provisions of any Act regulating the powers and duties of justices of the peace for such district, or of the police of such district; and with respect to other parts of Ireland, before a justice or justices of the peace sitting in petty sessions, subject and according to the

provisions of "The Petty Sessions (Ireland) Act, 1851," and any Act amending the same.

Every penalty herein imposed may be reduced or mitigated according to the judgment of the justices.

21. At the hearing of the information in such proceeding the production of the certificate of the public analyst shall be sufficient evidence of the facts therein stated, but the certificate may be supplemented by the personal evidence of the public analyst, and that of experts or other witnesses. If the defendant shall so require it, and give due notice of the fact, the public analyst shall be called as a witness. When the presence of the public analyst is required by the defendant, reasonable remuneration and expenses shall be allowed.

At the hearing of the case the part of the article retained by the person who purchased it, or which was returned to him by the public analyst, shall be produced. The defendant may, if he think fit, tender himself and wife to be examined on his behalf, and he or she shall, if he so desire, be examined accordingly.

No certificate, other than that of the public analyst, shall be received in evidence, unless supported by the oral evidence of the analyst giving the certificate.

22. In the event of conflict of evidence in any proceedings under this Act, the justices before whom any complaint may be made, or the court before whom any appeal may be heard, may, upon the request of either party, in their discretion cause any article of food or drug to be sent to the Commissioners of Inland Revenue, who shall thereupon direct the chemical officers of their department to analyse the same, and give a certificate to such justices of the result of the analysis. The chemical officers of Somerset House shall state in their certificates whether the article, in their opinion, is genuine or adulterated, or is, or is not, of the nature, substance or quality of the article indicated in the summons, and whether their analysis is in substantial agreement with that of the public analyst, and whether at the time they made the analysis the article was in such a condition as to allow of the results obtained by its analysis to be fairly comparable with those obtained by the public analyst. And the justices before whom the case may be heard may, at the request of either party, require the attendance of the officers of Somerset House who made the analysis, to give evidence in explanation of their certificate, and shall give a decision with the whole of the evidence before them. And the expense of such reference analysis and of the attendance of the chemical officers of the Inland Revenue Department, if required, shall be paid by the plaintiff or the defendant as the justices may by order direct.

23. Any person who has been convicted of any offence punishable by any Act hereby repealed or by this Act by any justices may appeal in England to the next general or quarter sessions of the peace which shall be held for the city, county, town, or place wherein such conviction shall have been made, provided that such person enter into a recognisance within three days next after such conviction, with two sufficient sureties, conditioned to try such appeal, and to be forthcoming to abide

the judgment and determination of the court at such general or quarter sessions, and to pay such costs as shall be by such court awarded; and the justices before whom such conviction shall be had are hereby empowered and required to take such recognisance; and the court at such general or quarter sessions are hereby required to hear and determine the matter of such appeal, and may award such costs to the party appealing or appealed against as they or he shall think proper.

In Ireland any person who has been convicted of any offence punishable by this Act may appeal to the next court of quarter sessions to be held in the same division of the county where the conviction shall be made by any justice or justices in any petty sessions district, or to the recorder at his next sessions where the conviction shall be made by the divisional justices in the police district of Dublin metropolis, or to the recorder of any corporate or borough town when the conviction shall be made by any justice or justices in such corporate or borough town (unless when any such sessions shall commence within ten days from the date of any such conviction, in which case if the appellant sees fit, the appeal may be made to the next succeeding sessions to be held for such division or town), and it shall be lawful for such court of quarter sessions or recorder (as the case may be) to decide such appeal, if made in such form and manner and with such notices as are required by the Petty Sessions Acts respectively hereinbefore mentioned as to appeals against orders made by justices at petty sessions, and all the provisions of the said Petty Sessions Acts respectively as to making appeals and as to executing the orders made on appeal, or the original orders where the appeals shall not be duly prosecuted, shall also apply to any appeal made under this Act.

24. In any prosecution under this Act, where the fact of an article having been sold in a mixed state has been proved, if the defendant shall desire to rely upon any exception or provision contained in this Act, it shall be incumbent upon him to prove the same.

25. If the defendant in any prosecution under this Act prove to the satisfaction of the justices or court that he had purchased the article in question in nature, substance, and quality as that demanded of him by the prosecutor, and with a written warranty to that effect, that he had no reason to believe at the time when he sold it that the article was otherwise, and that he sold it in the same state as when he purchased it, proceedings shall be taken against the giver or givers of the warranty, and for the purposes of this Act the offence or alleged offence shall be deemed to have been committed within the jurisdiction of the court hearing the original summons against the vendor. And if it should be proved to the satisfaction of the justices that the article in question was incorrectly described by the warrantor, the person or persons who issued, gave, or uttered such warranty shall be guilty of an offence under this Act, and be liable to a penalty not exceeding fifty pounds, and in the event of the conviction of the giver or givers of the warranty, but not otherwise, the original defendant shall be discharged from the prosecution, but shall be liable to pay the costs incurred by the prosecution, unless he shall have given due notice to the prosecutor that he would rely on the defence of warranty.

Provided that no such defence under this section shall be admitted, unless the

defendant prove that the giver of the warranty is resident and trading within the United Kingdom.

The term "warranty" in this Act shall include any written or printed document, invoice or label, purporting to be a warranty, invoice, or descriptive label, but shall not include a mere verbal declaration.

In any proceedings against the giver or givers of any warranty under this section, it shall be no defence to plead that action was not taken within twenty-eight days from the time of the purchase of the sample, but in all cases action shall be taken within a reasonable time.

26. Every penalty imposed and recovered under this Act shall be paid in the case of a prosecution by an officer, inspector, or constable of the authority who shall have appointed an analyst or agreed to the acting of an analyst within their district, to such officer, inspector, or constable, and shall be by him paid to the authority for whom he acts, and be applied towards the expenses of executing this Act, any Statute to the contrary notwithstanding; but in the case of any other prosecution the same shall be paid and applied in England according to the law regulating the application of penalties for offences punishable in a summary manner, and in Ireland in the manner directed by the Fines Act, Ireland, 1851, and the Acts amending the same.

27. Any person who shall forge, or shall utter, knowing it to be forged for the purposes of this Act, any certificate or any writing purporting to contain a warranty, shall be guilty of a misdemeanour, and be punishable on conviction by imprisonment for a term not exceeding two years with hard labour.

Every person who shall wilfully apply to an article of food or a drug in any proceedings under this Act a certificate or warranty given in relation to any other article or drug, shall be guilty of an offence under this Act, and be liable to a penalty not exceeding fifty pounds.

Every person who shall give a false warranty in writing to any purchaser in respect of an article of food or a drug sold by him as principal or agent, shall be guilty of an offence under this Act, and be liable to a penalty not exceeding fifty pounds.

And every person who shall wilfully give a label with any article sold by him which shall falsely describe the article sold, shall be guilty of an offence under this Act, and be liable to a penalty not exceeding fifty pounds.

28. Nothing in this Act contained shall affect the power of proceeding by indictment, or take away any other remedy against any offender under this Act, or in any way interfere with contracts and bargains between individuals, and the rights and remedies belonging thereto.

Provided that in any action brought by any person for a breach of contract on the sale of any article of food or of any drug, such person may recover alone or in addition to any other damages recoverable by him the amount of any penalty in which he may have been convicted under this Act, together with the costs paid by him upon such conviction and those incurred by him in and about his defence

thereto, if he prove that the article or drug the subject of such conviction was sold to him as and for an article or drug of the same nature, substance, and quality as that which was demanded of him, and that he purchased it not knowing it to be otherwise, and afterwards sold it in the same state in which he purchased it; the defendant in such action being nevertheless at liberty to prove that the conviction was wrongful, or that the amount of costs awarded or claimed was unreasonable.

EXPENSES OF EXECUTING THE ACT.

29. The expenses of executing this Act shall be borne, in the city of London and the liberties thereof, by the consolidated rates raised by the Commissioners of Sewers of the city of London and the liberties thereof, and in the rest of the metropolis by any rates or funds applicable to the purposes of the Act for the better local management of the metropolis, and otherwise, as regards England, in counties by the county rate, and in boroughs by the borough fund or rate;

And as regards Ireland, in counties by the grand jury cess, and in boroughs by the borough fund or rate, all such expenses payable in any county out of grand jury cess shall be paid by the treasurer of such county; and

The grand jury of any such county shall, at any assizes at which it is proved that any such expenses have been incurred or paid without previous application to presentment sessions, present to be raised off and paid by such county the moneys required to defray the same.

BOARD OF REFERENCE.

30. There shall be appointed a board or committee, consisting of the chief chemical officer of the Inland Revenue Laboratory, a person nominated by the General Medical Council, three persons, being public analysts, nominated by the Local Government Board, and a person nominated by the Board of Agriculture. These appointments shall lapse triennially except that of the chief chemical officer of the Inland Revenue Laboratory, but the same persons shall be eligible for reappointment.

This Board of Reference shall be paid such remuneration as shall be decided by the Local Government Board, in consultation with the Board of Agriculture and the Treasury, and their duty shall be from time to time to examine and report on the composition of various articles of food and drugs, and to describe, investigate, and devise new or improved methods for their examination, to set forth definitions and exceptions, and to fix limits and standards of quality and purity.

The recommendations, definitions, exceptions, limits, and standards of the Board of Reference shall, on ratification by Order in Council and publication in the *London Gazette*, be binding in law, and shall continue in force until such Order in Council be rescinded, and the said committee shall meet together at least times annually for the purpose of consultation, and shall issue from time to time such new or revised regulations, definitions, exceptions, limits, and standards as they in their discretion may deem desirable.

SPECIAL PROVISION AS TO TEA.

31. From and after the first day of January, one thousand eight hundred and seventy-six all tea imported as merchandise into and landed at any port in Great Britain or Ireland shall be subject to examination by persons to be appointed by the Commissioners of Customs, subject to the approval of the Treasury, for the inspection and analysis thereof, for which purpose samples may, when deemed necessary by such inspectors, be taken, and with all convenient speed be examined by the analysts to be so appointed; and if upon such analysis the same shall be found to be mixed with other substances or exhausted tea, the same shall not be delivered unless with the sanction of the said Commissioners, and on such terms and conditions as they shall see fit to direct, either for home consumption or for use as ships' stores or for exportation; but if on such inspection and analysis it shall appear that such tea is in the opinion of the analyst unfit for human food, the same shall be forfeited and destroyed or otherwise disposed of in such manner as the said Commissioners may direct.

32. Tea to which the term "exhausted" is applied in this Act shall mean and include any tea which has been deprived of its proper quality, strength, or virtue by steeping, infusion, decoction, or other means.

33. For the purposes of this Act every liberty of a cinque port not comprised within the jurisdiction of a borough shall be part of the county in which it is situated, and subject to the jurisdiction of the justices of such county.

34. Every liberty having a separate court of quarter sessions, except a liberty of a cinque port, shall be deemed to be a county within the meaning of this Act.

35. The town council of any borough having a separate court of quarter sessions shall be exempt from contributing towards the expenses incurred in the execution of this Act in respect of the county within which such borough is situate, and the treasurer of the county shall exclude the expenses so incurred from the account required by section one hundred and seventeen of the Municipal Corporation Act, 1835, to be sent by him to such town council.

36. The town council of any borough having under any general or local Act of Parliament, or otherwise, a separate police establishment, and being liable to be assessed to the county rate of the county within which the borough is situate, shall be paid by the justices of such county the proportionate amount contributed towards the expenses incurred by the county in the execution of this Act by the several parishes and parts of parishes within such borough in respect of the rateable value of the property assessable therein, as ascertained by the valuation lists for the time being in force.

37. In the application of this Act to Scotland the following provisions shall have effect:

1. The term "misdemeanour" shall mean "a crime or offence";
2. The term "defendant" shall mean "defender," and include "respondent";

3. The term "information" shall include "complaint";
4. This Act shall be read and construed as if for the term "justices," wherever it occurs therein, the term "sheriff" were substituted;
5. The term "sheriff" shall include "sheriff substitute";
6. The term "borough" shall mean any royal burgh and any burgh returning or contributing to return a member to Parliament;
7. The expenses of executing this Act shall be borne in Scotland, in counties, by the county general assessment, and in burghs by the police assessment;
8. This Act shall be read and construed as if for the expression "the Local Government Board," wherever it occurs therein, the expression "one of Her Majesty's Principal Secretaries of State" were substituted;
9. All penalties provided by this Act to be recovered in a summary manner shall be recovered before the sheriff of the county in the sheriff court, or at the option of the person seeking to recover the same in the police court, in any place where a sheriff officiates as a police magistrate under the provisions of "The Summary Procedure Act, 1864," or of the Police Act in force for the time in any place in which a sheriff officiates as aforesaid, and all the jurisdiction, powers, and authorities necessary for this purpose are hereby conferred on sheriffs;

Every such penalty may be recovered at the instance of the procurator fiscal of the jurisdiction, or of the person who caused the analysis to be made from which it appeared that an offence had been committed against some one of the provisions of this Act;

Every penalty imposed and recovered under this Act shall be paid to the clerk of court, and by him shall be accounted for and paid to the treasurer of the county general assessment, or the police assessment of the burgh, as the sheriff shall direct;

10. Every penalty imposed by this Act may be reduced or mitigated according to the judgment of the sheriff;
11. It shall be competent to any person aggrieved by any conviction by a sheriff in any summary proceeding under this Act to appeal against the same to the next circuit court, or where there are no circuit courts to the High Court of Justiciary at Edinburgh, in the manner prescribed by such of the provisions of the Act of the twentieth year of the reign of King George the Second, chapter forty-three, and any Acts amending the same, as relate to appeals in matters criminal, and by and under the rules, limitations, conditions, and restrictions contained in the said provisions.

SCHEDULE I.

Form of Certificate.

To*

I, the undersigned, public analyst for the _____, do hereby
 certify that I received on the _____ day of _____, 18____, from†
 _____, a sample of _____ for analysis
 and have analysed the same, and declare the result of my analysis to be as follows :
 I am of opinion that

Observations.‡

As witness my hand this

day of _____,

A. B.,

at _____.

* Here insert the name of the person submitting the article for analysis.

† Here insert the name of the person delivering the sample, when known, or agency through which it was received.

‡ Here the analyst may insert at his discretion his opinion as to whether the mixture (if any) was for the purpose of rendering the article portable or palatable, or of preserving it, or of improving the appearance, or was unavoidable, and may state whether in excess of what is ordinary, or otherwise, and whether the ingredients or materials mixed are, or are not, injurious to health.

In the case of a certificate regarding any article liable to decomposition, the analyst shall specially report whether any change had taken place in the constitution of the article that would interfere with the analysis.

SCHEDULE II.

List of articles bearing a name identical with British Pharmacopœia remedies,
 but not required to be of B.P. quality.

SCHEDULE III.

Official definitions, limits, and standards of strength and purity adopted by the
 Board of Reference.

Spirits, as at present defined by the Amendment Act; milk; water in butter;
 lard; cheese; vinegar; impurities in tea, pepper, and other spices; drugs.

THE ANALYST.

AUGUST, 1894.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

PRELIMINARY NOTES ON THE COLORIMETRIC ESTIMATION OF MINUTE QUANTITIES OF LEAD, COPPER, TIN, AND IRON.

BY EDWARD RUSSELL BUDDEN AND HERBERT HARDY.

(Read at the Meeting, April 4, 1894.)

WE frequently have occasion to examine samples of liquids containing traces of metals derived from impurities in the ingredients employed in their manufacture, or now more frequently arising from solution of metallic surfaces of machinery with which the liquids come in contact in process of preparation. We may say that in the latter case we find strong evidence in support of our view that the nature and causes of such metallic contamination have hitherto not received sufficient consideration at the hands of many well-known analysts, and have, indeed, in our opinion been in several important cases (of course, quite unintentionally) mis-stated.

There can be very little doubt that some modified gravimetric process would be preferable in many cases of examination of samples of mineral waters and allied aerated beverages. A process of this nature was suggested and carried out with admirable results some time since in cases in which we had the privilege to be associated with Mr. Otto Hehner. But it frequently happens that the analyst is compelled by smallness of sample, or other equally potent reasons, to adopt some process involving a smaller expenditure of material and time. We may, perhaps, be allowed to say incidentally that in our opinion the *time* occupied in commercial analysis does not always receive its due recognition, and it seems very desirable that there should be more uniformity in fees for different classes of work. But, be that as it may, as a matter of fact it is often necessary to employ some form of volumetric process.

The method most frequently adopted appears to be that originally suggested by Miller, in which the solution (rendered acid, or alkaline, according to the class of metal required to be estimated) has a certain quantity of sulphuretted hydrogen in aqueous solution added, and is then compared with a solution containing a known quantity of the same metal similarly treated. The process is, of course, closely comparable with the Nessler test, familiar to every water-analyst, and it gives fairly good results if only one metal be present. In many laboratories a very small quantity of ammonium sulphide solution is used in place of the sulphuretted hydrogen solution, the test being made in the manner suggested by Wanklyn.

Although we find that this method possesses some advantages (in the case of water-analysis, for example), we have, nevertheless, found many practical disadvantages attending its use in some other classes of work, notably in the case of

liquids possessing a slight yellow tinge in the first instance. And, further, we find no little difficulty in attaining uniform tint in the porcelain dishes in which this test is usually conducted.

We have, therefore, adopted a form of Nessler glass for tests of this description, and invariably use freshly-prepared sulphuretted hydrogen solution. We also find it almost essential for obtaining really accurate results to adopt some form of screen which allows white light to pass vertically only through the Nessler cylinder. We had some difficulty at first in obtaining glasses of uniform colour. But by selecting (by kind permission of a firm of manufacturers) glasses from a large stock, we obtained some few sufficiently good for ordinary purposes.

As we found that some curiously contradictory results were occasionally obtained, we were led to note carefully the conditions under which uniformity was possible. Of course, such results would only be noticed by those whose position brought into the laboratory a very large number of samples of one class of goods.

As the result of these observations, we thought it desirable to make a large number of experiments under varying conditions, and we have the honour to lay before the society a very brief epitome of some of the results obtained, in the hope that new suggestions may be made, and better methods of work suggested. For papers are only of value when they induce fresh investigation and improve our common work. If it be wished, we can give some of our work in greater detail, but it seems to us undesirable to over-burden the journal with excessive detail, or to make papers for the purpose of attracting attention to individual workers.

For purposes of comparison, we prepared standard solutions of such strength that 1 cubic centimetre contained 0.0001 gramme of metal. The method of making the test was as follows:

An empirical solution of any given metal was made, and a certain amount added to filtered distilled water in a Nessler tube, and made up to exactly 50 c.c. with similar water; then a few drops of acetic acid were added, and finally about 2.5 c.c. of a fresh saturated solution of sulphuretted hydrogen in water.

The same process was carried out with the substitution of the standard metal solution (in the proportion requisite for giving a precisely similar tint) for the empirical metallic solution, and the strength of the latter thus determined.

But in the case of the standard solution, the sulphuretted hydrogen solution was sometimes added to the water before the addition of the metallic solution, and at other times the metallic solution was added before the introduction of the sulphuretted hydrogen. It was found that a very important difference arose in the two different orders of addition. For instance, in the experiments with an unknown quantity of lead acidulated with acetic acid, to which sulphuretted hydrogen was *subsequently* added, it was found that when the attempt was made to produce the same tint by adding the standard lead solution to the water *already* mixed with the requisite amount of sulphuretted hydrogen the difference amounted to 0.1 c.c. and upwards in the quantity required. Though it was found that in the case of very dilute lead solutions fairly uniform results could be obtained.

Experiments of a similar character with copper gave also very discordant results. For instance, in three successive tests 3.5 c.c., 4.0 c.c., and 4.2 c.c. were

respectively used to secure similarity of tint, giving a difference of 0.7 c.c. between extremes, arising from difference of order of addition of reagents.

We next passed on to experiments with mixtures of metals, since it often happens that several metals are present in fluids with which we are practically concerned. We may mention at this point the value which we found in Dr. Teed's suggestion (made in a paper read before the society some time since), that the coloration, due to the presence of copper, might be prevented by addition of potassium cyanide, while, when iron also is present, its influence on colour reactions may be avoided by the employment of tartaric acid. It is, perhaps, hardly needful to say that this latter must be absolutely pure, and that the tests must be made in alkaline solution.

Certain difficulties, however, arise when other metals are present, to which we shall have occasion to refer subsequently, although Dr. Teed's method possesses great value in many cases.

For experiments on mixed metals we employed solutions containing varying proportions of standard metallic solutions, as we had reason to believe that difficulty would arise in the direction of tints of matching solutions. For it should be noted that in the case, for instance, of lead and copper the tint given in each case by the addition of sulphuretted hydrogen differs widely. In fact, it is practically impossible to carry out the volumetric process with mixtures of some metals on account of the complexity of tints.

We first experimented with copper and lead in the following way: Taking a mixture of equal quantities of solutions containing approximately the same amount of lead and copper, we found it impossible to match the colour accurately either with standard copper or standard lead solution alone, though some approximate estimate could be formed of the amount of metal present. When copper was kept in solution, however, by addition of potassium cyanide, and the mixture matched by standard lead solution alone (still adding the metal after the introduction of the sulphuretted hydrogen, as in the case of our experiments with single metals), we found in some cases an error amounting to 0.2 c.c. of standard metal solution, or about 20 per cent.

Thus it will be seen that adding the standard metallic solution after the sulphuretted hydrogen to the matching solution in the Nessler cylinder is a method capable of introducing most serious errors into the determination. The nature of the difficulty is apparently indicated by our observation that on using rather stronger solutions than we usually employed, we noticed the production of a minute film of metallic sulphide upon the surface of the liquid as the metallic solution was run in from the burette. This effect is specially noticeable in the case of lead. The production of this film, of course, has the effect of causing more standard metallic solution to be employed than is actually required in any given experiment. For example, in one experiment we recorded an error of 2 c.c. (6 c.c. required in place of 4 c.c.), which equals an experimental error of 50 per cent. of the actual amount of metal present. We also observe that the error increases materially with stronger solutions.

A mixture of lead and copper can be better estimated by employing for com-

parison a lead solution than one of copper, while in this case the addition of potassium cyanide appears to cause too little lead to be used for matching if the metal be added to the solution after the sulphuretted hydrogen. Though in this case the error does not exceed 10 or 20 per cent., yet such an amount seriously impairs accuracy.

It does not appear to make any difference whether lead be compared in acid or alkaline solutions if no other metal be present.

For estimation of mixtures of lead and copper we tried compound matching solutions containing the two metals in equal and other proportions, but found but little success in this direction. Although the difference in colour between the tubes was slight, it was very difficult to judge correctly, while if the proportions of metals are very unequal the difficulty is much increased. If the metal be added before the sulphuretted hydrogen, there is no difficulty in obtaining closely concordant results in the case of copper alone, lead alone, or a mixture of lead and copper. In the case of a mixture of this kind, if potassium cyanide be added to keep up the copper, and the sulphide tint be matched with standard lead, and in a following experiment (on a similar quantity of liquid) an amount of standard lead be first added equal to that used in the first instance, and the difference again matched, using this time standard copper, very accurate results may be obtained, provided always that the metal be added before the addition of sulphuretted hydrogen, otherwise altogether conflicting results are obtained, which are entirely worthless.

It seems to be essential that all experiments with this method must be made under strictly comparable conditions. The same amount of each reagent must be used in each case, and the liquid should be well stirred after each reagent has been added.

The results seem to be most correct when the sample is of the same strength so far as metal is concerned, as the standard metallic solution.

There is a slight error noticeable if much difference exists between the strengths of the solutions, but this error is too small to affect general accuracy of results in practice. If iron be present, it may be prevented from interfering with the tint reactions by the addition of lead-free tartaric acid (as suggested by Dr. Teed).

In those cases where potassium cyanide has also been added to keep the copper in solution, there may be formed a precipitate of Prussian blue if much iron be present. In such an event it would probably be better to estimate the iron by one of the less delicate ordinary volumetric methods.

Thus we have indicated how copper and iron can be practically eliminated from the colour-test and lead estimated in the presence of these metals with considerable accuracy. But in almost every sample of artificial mineral waters, we find that a very notable proportion of tin is discernible. The reason for this and other contamination we shall speak of presently.

We find that by the use of peroxide of hydrogen, tin (if present in small quantities only) can be completely and rapidly oxidized, and will then not perceptibly affect the colorimetric method. But the test must be made rapidly, since after about a minute there is a slight colour perceptible, especially if the amount be somewhat larger than usual; and the colour speedily deepens, and after about five minutes the solution becomes opaque.

An amount of tin that in the stannous condition gives with sulphuretted hydrogen a blackness that makes it impossible to see through two inches of solution, will, if oxidized with peroxide of hydrogen, give, on addition of sulphuretted hydrogen, no more colour than can be matched by less than 0.1 c.c. of standard lead solution. But it becomes opalescent after a very short time.

A standard stannous solution (0.001 gramme tin per c.c.) may be employed for matching the tint accurately, but this will not keep for more than two days. When tin is present as well as lead or copper, the matching with lead or copper or a mixture of both becomes impossible. Therefore the tin must be prevented from affecting the results in some manner such as we have suggested; otherwise large errors of analysis will arise. It therefore seems to us possible to test accurately, on the lines we have indicated, a complex mixture of the four metals most commonly found in combination. But we find that to give correct and comparable results, all tests must be made under similar conditions. The reagents must be added in the same proportion and same order. The sulphuretted hydrogen solution must be added after the addition of metallic solution in the comparison cylinder, otherwise a scum of sulphide is formed seriously affecting results. It may affect them to the extent of 50 per cent. Also, if the metal be added after the sulphuretted hydrogen, a clear tint cannot readily be obtained, while if the addition be made in the proper order, a clear and readily-matched tint is obtained.

The use of potassium cyanide appears sometimes to give rise to complications, owing to the formation of complex and unstable cyanogen compounds, especially in solutions containing iron. It also seems probable that when a large proportion of copper is present a portion may be precipitated after the addition of potassium cyanide. Possibly this may be due to the formation of a cupro-potassium cyanide in which a portion of the metal is capable of precipitation as sulphide.

Peroxide of hydrogen completely destroys the colour produced by some of the metallic sulphides, even when the tints are very strong. But there is no sharp transition from the oxidation of one sulphide to that of another, though the changes appear to take place in some definite order. Yet the stages are too transient to admit of any satisfactory conclusion being drawn from noting the progress of this reaction, otherwise some valuable information as to the relative proportion of the metallic impurities might possibly have been deduced.

In the case of aerated beverages there is always present a notable amount of carbonic acid gas. It therefore seemed desirable to ascertain if any marked effects were produced upon the tints formed in the reactions employed for determining traces of metallic contamination. Experiments were made, therefore, on this point with rather curious results, which may tend to explain certain anomalies hitherto unaccounted for. The gas was in some instances passed through the solutions before the addition of reagents, and sometimes afterwards. Stated briefly, the experiments hitherto made in this direction show that the iron reactions are most influenced by the presence of carbonic acid; that iron is the only metal affected when the gas is passed first, *i.e.*, before any colour-reaction is produced, and this has an important bearing on the testing of mineral waters. Further, it would seem that copper is never affected by the gas. When the colour reaction has taken place with lead and tin,

the passing of carbonic acid gas causes the sulphides to assume the form of a precipitate.

In a paper of great value read before the Society of Chemical Industry by Mr. R. Warrington, some observations were made on the employment of glycerol in connection with the sulphide tests for lead in tartaric and citric acids. We therefore made a few experiments to judge of the effect of this addition in the case of other metals, and obtained one or two results of interest.

The addition of a small amount (two or three drops) of glycerin partially prevents the formation of a colour or precipitate in the case of all the metals. The employment of 5 c.c. in 50 c.c. of solution does not materially increase this effect, while if a volume of glycerine equal to that of the tested solution be employed, the precipitation is entirely prevented, even in the case of iron, which under the ordinary conditions obtaining in these experiments was found to precipitate most readily. It seems probable that this curious effect of glycerin is due to mechanical rather than chemical causes, but it would seem to indicate the necessity of tests being made in solutions of similar density if really accurate results are desired.

As the result of experiments and observations hitherto made, we deduce the following conclusions:

That it is above all necessary in determination of traces of metals made by these volumetric processes to follow strictly the same order in the addition of reagents, and as closely as possible to employ always the same quantity of reagent, both in the actual experiment and in the preparation of the solution used for the comparative determination.

That all the conditions of experiment must be similar, for example, as to the state of oxidation of the metal, and the presence of extraneous factors affecting reactions, as in the case of carbonic acid gas or substances affecting specific gravity of samples tested. It would seem that, if our results are confirmed, these experiments carry the method of testing for metallic impurities in solution somewhat further than has hitherto been possible, but we are still hopeful of working out a more completely successful process of differential volumetric testing in the case of solutions containing several metals. If this can be accomplished, much facility will be afforded to the analyst who desires to obtain quickly and accurately an estimation of the proportions of metals causing the aggregate metallic contamination of the beverage or solution submitted for examination.

We may observe that in the case of aerated waters and similar beverages artificially prepared, the predominant metal appears in all cases to be tin. We are speaking, of course, of the products of well-appointed manufactories, equipped with modern machinery, and in which only pure acids are employed. Formerly, when it was practically impossible to obtain tartaric and citric acids free from lead, that metal was frequently found in lemonade and ginger-beer, and other acidulated beverages; while many cases have been recorded, usually from small and imperfectly equipped factories, where a serious amount of lead or copper contamination arose from the use of unsuitable pipes or other appliances. Mr. Stokes showed one of us a sample of ginger-beer from such a source containing a very considerable amount of lead (about 4.5 grains to the gallon). And sometimes contamination has arisen from

the glazing of stoneware bottles and other appliances, such as enamelled metal pans used sometimes for syrup-making. Copper also finds its way from machinery which has not been perfectly coated with some non-injurious material; and there is reason to believe that in some cases, at any rate, a very appreciable amount of copper is dissolved in the aerating cylinders by an electrolytic action set up by the dissolved carbonic acid coming into contact with the tin lining of the vessels and portions of copper exposed by continuous friction. Indeed, we have found a marked increase of copper dissolved when a cylinder has been (by request) allowed to remain filled with carbonated water for an unusually long period. All these points have to be taken into consideration when forming an opinion as to the cause and nature of metallic contamination of beverages. But the most important matter for the analyst is usually to be able to rapidly and accurately estimate the extent of total metallic impurity, and when the quantity is at all considerable, to have a ready method of determining the nature and proportion of the metals severally concerned in producing such total. If these notes tend in any degree to clear the ground in this direction, they will not have unduly occupied the time of the society, we trust.

DISCUSSION.

The Chairman (Mr. O. Hehner) invited discussion on the subject of the paper, and said that he hoped that this would lead to some agreement as to a method for testing water for traces of metallic impurities.

Dr. J. Edmunds referred to the pyrological methods introduced by Colonel Ross, and improved by himself, for the detection and estimation of exceedingly minute quantities of metals. These consisted in reducing a portion of the ash to be examined in a bead of pure boric acid in the flame of the blowpipe, and subsequently examining and measuring the spherule of reduced metal, after the bead had been mounted in Canada balsam, under the microscope. By the use of the filar micrometer, the diameter of the spherules might be taken with great accuracy, and the geometric constants—used with the chemical formula and specific gravity of the substance of the spherule—gave its weight to the millionth of a gramme or even less. A spherule from $\frac{1}{1000}$ to $\frac{1}{10000}$ of an inch in diameter was an easy object to deal with by this method. Copper and lead could be distinctly dealt with in this way, but he (Dr. Edmunds) had not been able to satisfy himself that the method was reliable for tin. Lead and some other metals might be got out on a platinum cathode under the slow action of a couple of Daniell's cells, and then dissolved off and dried up to a residue fit for treatment in boric acid.

Mr. Sidney Harvey thought it was imperatively necessary that the same order should be observed in the comparison of the liquids with the trial test in the case of the estimation of small amounts of metal in solution. That is to say, the reagents must be added in the same order, and in the same amounts.

Dr. Frank L. Teed believed that the amount of lead in a water, when it amounted to one-hundredth of a grain to the gallon, could only be determined by some volumetric process. He had listened with great interest to what Dr. Edmunds had said, but he looked upon his method as a qualitative rather than a quantitative

method of analysis. Mr. Budden's remarks were extremely important, more especially as regards the order in which the solutions were added. In a paper on the same subject Mr. Warington had pointed out that citric and tartaric acids had a specific action on the tint of the lead coloration. He had certainly shown that lead and copper assumed different tints according as they were in presence of water only, or of tartaric, or of citric acid. At the time he (Dr. Teed) was working out his process (on which Mr. Warington's was subsequently based), he was stopped by this obstacle, that there was no pure tartaric, and no pure citric acid to be obtained for love or money. He tested a good many samples, but they invariably contained lead. He suggested in his paper that for the separation of copper, lead, and iron in a natural water, pure tartaric acid should be used, but as he had not got any he could not say that he ever did it. Mr. Warington had proved that citric and tartaric acid affected the colour, and Mr. Budden had shown that carbonic acid also affected it. If it was desired to determine the presence of lead in ginger-beer, he believed the only possible way was to ash it first and get rid of the organic matter. He hoped that Mr. Budden would bring before the society more elaborate notes later on, so that there might be some standard of reference in regard to this subject. In his opinion, when it was a question of .0003 per cent. of lead in a sample, it did not matter whether the result was .0003 or .0004.

Mr. E. J. Bevan did not know whether Dr. Teed made it quite clear to what extent Mr. Warington looked upon the presence of citric acid as the cause of error. He (Mr. Bevan) had had occasion to work on a large number of beverages some time ago, and he had some conversation with Mr. Warington on the subject, and that gentleman informed him of the results he intended to bring forward. He (Mr. Bevan) had found that by adding sugar to the solution the apparent amount of lead was increased as much as ten times. It was necessary, in order to make a satisfactory colorimetric test, that the disturbing constituents should be known, and these must be added to the standard solution. Dr. Teed's course of evaporating down and then igniting would get over that difficulty. It was a remarkable thing that an error of 1,000 per cent. could take place if sugar were present.

Mr. Hehner asked Mr. Budden how he used the peroxide of hydrogen. He understood him to say that turbidity very soon manifested itself.

Mr. Budden replied that he added the peroxide of hydrogen and subsequently the sulphuretted hydrogen water.

Mr. Richmond drew attention to the work of Messrs. Picton and Linder (*Jour. Chem. Soc.*, lxi., 114, 137, and 148), which probably contained the whole explanation of the curious differences observed by Mr. Budden. It seemed to him that variations of colour were due, in some cases, to the formation of different hydro-sulphides. Thus, Messrs. Picton and Linder had obtained with copper hydro-sulphides approximating to the formulæ $7\text{CuS}, \text{H}_2\text{S}$; $9\text{CuS}, \text{H}_2\text{S}$; and $2\text{CuS}, \text{H}_2\text{S}$, of which the first, obtained with excess of sulphuretted hydrogen, was soluble. It seemed to him (Mr. Richmond) that the results of Mr. Warington with citric acid, and those of Mr. Bevan with sugar, were explained by the second and third papers of Messrs. Picton and Linder; they found that different physical modifications show different

colours, and the exact state in which they exist depends partly on the other constituents of the solution. Possibly in these cases the sulphides were more in suspension than they would be with water only as a solvent, and the presence of substances such as citric acid and sugar turned the dissolved body out of solution into a condition of suspension, and thus made the tint appear darker; and therefore in testing for these metals the best thing to do would be to have the conditions most favourable for a state of suspension as against a state of solution—that is to say, this condition should be pushed as far as was compatible with the non-production of actual precipitation, in order to develop the maximum coloration. A study of these papers would be found very interesting.

Mr. Hehner said he believed the statement that citric acid added in quantity would make these sulphides more insoluble was inaccurate. His experience was that citric acid had a contrary effect.

Mr. Richmond admitted that Mr. Hehner's remark, that citric acid prevented complete precipitation of lead sulphide, certainly seemed an apparent contradiction to what he had quoted from Messrs. Picton and Linder's work. But he thought that if their work were read carefully, it would be seen that they had shown that there were so many stages between the actual precipitation and true solution that he (Mr. Richmond) did not think it would be entirely in contradiction. He had also found that lead precipitated as sulphide in presence of citric acid when filtered would run through the filter; but, notwithstanding, though the particles could hardly be seen, the sulphide could be separated from the solution fairly easily, and it certainly was in a state of suspension.

Mr. Bevan asked Mr. Budden what the action of glycerin was; did it keep the lead in solution?

Mr. Budden replied that if there was a certain amount of glycerin added precipitation was prevented, and the matter was kept in solution.

Mr. Hehner thought that the deductions to be drawn from the discussion showed that small quantities of these metals, when mixed together, could be quantitatively determined under certain conditions by colour-tests. When they were in simple aqueous solution there was generally no difficulty; but when it was shown that under other conditions an error might be committed, faith in the colorimetric process was shaken. He had refused to make determinations on very minute quantities of the sample; but he had attempted, with more or less success, by taking a large quantity and evaporating to small bulk, to separate the metals. He imagined that it would be impossible to obtain the metals by incineration. The quantities of sugar and the carbon obtained therefrom were very large. One could not expect to incinerate a milligramme of lead with ten thousand times its weight of sugar without a considerable loss occurring. He would like to ask Mr. Budden whether he had ever used a colorimeter, such as Mills', which would obviate diluting or adding more reagent to the liquid. It was very desirable to arrive at some plan of operation for the future.

Mr. Budden said that the reason which prompted him to bring forward his paper was because he found that some manufacturers of aerated beverages were going the round of the London analysts, and were obtaining results from different chemists.

which disagreed with each other, and this seemed to him not only a very unsatisfactory state of affairs, but also one which was not calculated to impress the manufacturers (who wanted to know how they were to get rid of errors in manufacture) with a very high estimate of the methods used by analysts. In regard to ginger-beer, he might say that the metal which generally had to be looked for was lead, and when he could get the beverage sufficiently clear the addition of a trace of acetic acid, and then hydrogen sulphide in solution, enabled the colour to be observed with a certain amount of accuracy. He did not mean to say that it was a perfectly accurate method. The colour produced by ammonia when added to ginger-beer would, of course, necessarily interfere very much with the observations, but he did not see that in some cases it was necessary to have this solution alkalized to that extent. The ammonia appeared to react with some resinous matter in the ginger-beer. The point mentioned by Mr. Bevan interested him exceedingly, because it seemed to be almost the converse of the observation he (Mr. Budden) made about carbonic acid. He had had a case of some dozens of ginger-beer sent to him, all manufactured at one making. After standing some time, the contents of the bottles were found to vary materially in the reactions which they gave, and were also found to vary very materially in the amount of gas present. It seemed obvious that if the amount of effervescence was considerable in any given bottle, there must be a very marked difference in the amount of sugar present. It is very probable, indeed, that the sugar had a marked effect in the same way that glycerine had. He had been making some experiments on a form of colorimeter, and he had had an appliance made which might possibly be useful. It was certainly quite probable that the method might be considerably amplified.

THE EXAMINATION OF URINE FOR SMALL QUANTITIES OF SUGAR.

BY ALFRED H. ALLEN.

(Read at the Meeting, June 6th, 1894.)

THE detection and quantitative determination of sugar in typical diabetic urine presents few difficulties, since in such an excretion the proportion of sugar present is very considerable—ranging from 1 to 10 per cent.—and the interfering constituents of the urine are correspondingly reduced in quantity. But when the proportion of sugar is but small, by which I mean below 0.25 per cent., the difficulties attaching to its recognition and accurate determination are considerable, and have not been wholly overcome by any of the numerous investigators of the problem. Under these circumstances, it appeared probable that a review of the present position of the question, supplemented with some observations of my own, would be of interest to the members of the Society.

The question of the occurrence of traces of sugar in normal urine has been the occasion of much controversy. Brücke appears to have been the first to state that all normal urine contained sugar, and this view was supported by Bence Jones and by Kühne, but opposed by Friedlander, Wiederhold, Meissner, and Babo. The question was re-examined in 1871 by Seegen, who pointed out many fallacies in the methods of those who had found sugar in normal human urine, and concluded that it was either

absent, or present in such small proportion that the then existing methods were insufficient for its positive recognition in the presence of co-occurring bodies which simulate many of its reactions. On the other hand, Dr. F. W. Pavy, in 1878, concluded that sugar was a normal constituent of urine, and that no sharp line of demarcation could be drawn between the excretion in health and in diabetes, except quantitatively. Mr. G. Stillingfleet Johnson, again, by an improved method of examination found no trace of sugar in healthy urine, while Molisch, from the examination of a large number of samples of healthy human urine by the alpha-naphthol and thymol tests, came to the conclusion that traces of sugar are met with frequently in human urine; but the value of his tests, and hence the accuracy of his conclusions, have been disputed by Leuken and also by Seegen (*Jour. Soc. Chem. Ind.*, vi., 149, 150). E. Luther, again (*Chem. Centr.*, 1891, ii., 90; and *Jour. Chem. Soc.*, lx., 1559), as the result of the application of the furfuraldehyde and alpha-naphthol tests to a large number of samples, concludes that glucose is present in all human urine, the amount found in the excretion of adults averaging 0.1 per cent., while the total carbohydrates amount to 0.2 per cent. According to E. Roos (*Zeit. Physiol. Chem.*, xv., 513), the normal urine of the dog, horse, and rabbit always contains more or less carbohydrates, as indicated by the furfuraldehyde reaction and confirmed by the benzoic chloride test. Human urine is stated by Roos always to give an affirmative reaction with phenyl-hydrazine, and the same is true of dogs' urine, while the excretion of rabbits gives especially well-formed crystals. The urine of all these animals was found to be slightly lævo-rotatory.

By the production of the crystalline phenyl-glucosazone—a method free from the objections and fallacies which underlie nearly all other tests—I have proved to my complete satisfaction that sensible quantities of sugar are present in some specimens of urine from healthy persons.

The fact appears to be that, while normal human urine may sometimes contain traces of sugar, that substance is by no means constantly present, and a great number of the recorded observations are quite inconclusive.

Passing by the polarimetric and fermentation tests for sugar, neither of which is adapted for the recognition of very small quantities, the tests dependent on the reducing action of glucose are those which are best known and deservedly popular. But it is well known to all who have worked on the subject that these tests often give very disappointing results, even in the hands of experienced analysts, and when employed in the hurried and slovenly way very common in clinical testing give indications which are difficult to interpret or absolutely worthless. These failures, and the false conclusions frequently based on them, are largely due to the interfering action of some of the normal constituents of urine, the presence or influence of which is usually ignored. Hence it is important to consider the extent to which these bodies interfere, and the manner in which they may be removed or their influence obviated.

The chief of these interfering bodies are uric acid, xanthine, and creatinine, but under some conditions urine contains glycuronic acid or compounds thereof which simulate sugar very closely. Hence before the usual tests can be satisfactorily employed for the detection of small quantities of sugar, the above interfering sub-

stances should be removed as far as possible. The amount of uric acid passed per diem under ordinary conditions is said to be about 0.5 gramme, though, of course, in many instances it is considerably more. Xanthine and the allied bodies are present in still smaller amount. Although chemists are in the habit of taking account of uric acid, because it makes itself evident to the senses, they habitually ignore the presence of creatinine. According to Voit, the proportion of creatinine passed in twenty-four hours ranges from 0.5 to nearly 5 grammes. Urine containing the latter amount would exert a reducing action on Fehling's or Pavy's solution equivalent to the presence of 0.32 per cent. of glucose. Hence it is very important to remove these interfering bodies when testing for small quantities of sugar. For this purpose metallic precipitants can be used with considerable success. Thus, for instance, urine can be conveniently clarified and freed from albumen (if present), uric acid, phosphates, and colouring matters, by precipitating boiling hot with neutral lead acetate. Basic lead acetate removes certain other bodies which escape precipitation by the neutral salt, but there is no material advantage in its use. In either case, the filtered liquid is colourless, or very pale, and is well fitted for optical examination, or testing by the phenyl-hydrazine reaction.

Cupric sulphate yields little or no precipitate with normal urine in the cold, but on standing or boiling a whitish or pale-green precipitate is thrown down which has a tendency to darken if the heating be continued. If acetate of copper be substituted for the sulphate, or if sodium acetate be added to the cupric sulphate solution, the precipitation is much more complete, uric acid, xanthine, hypoxanthine, colouring matter, and albumen (if present) being entirely thrown down, and the creatinine and phosphates partially.* The filtered liquid cannot be used for the phenyl-hydrazine test, and the presence of copper unfits it for titration by Pavy's solution; but I have found it admirably suited for the detection of small quantities of sugar by Fehling's test, as follows:

From 7 to 8 c.c. measure of the sample of urine is heated to boiling in a test-tube, and, without separating any precipitate of albumen which may be thrown down, 5 c.c. of the solution of cupric sulphate used for preparing Fehling's test is added. This produces a precipitate containing uric acid, xanthine, hypoxanthine, phosphates, etc. To render the precipitation complete, however, it is desirable to add to the liquid, when partially cooled, from 1 to 2 c.c. of a saturated solution of sodium acetate having a feebly acid reaction. The liquid is next filtered. To the filtrate, which will have a bluish-green colour, 5 c.c. of the alkaline tartrate mixture used for preparing Fehling's solution is next added, and the liquid boiled for fifteen to twenty seconds. In the presence of more than 0.25 per cent. of sugar, separation of cuprous oxide occurs before the boiling-point is reached, but with smaller proportions precipitation takes place during the cooling of the solution, which becomes greenish, opaque, and suddenly deposits cuprous oxide as a fine orange-yellow precipitate. When a urine rich in sugar is under examination, the volume taken can be advantageously reduced from 7 or 8 c.c. to 2 or 3 c.c., or even less, water being added to replace it.

It is evident that in this modification of the ordinary Fehling's test, advantage

* The precipitation of the phosphates of urine by cupric acetate is not complete, but the circumstance has no influence on the detection of glucose in the filtrate.

is taken of the very general precipitating power of cupric acetate, to remove from the urine the great majority of those substances which interfere with the detection of diabetic sugar, by themselves reducing the alkaline copper solution, retaining the cuprous oxide in solution, or producing a flocculent precipitate which masks the true reaction of sugar. Operating as described above, no greenish turbidity refusing to settle is produced, and hence the separation of any cuprous oxide is very readily observed.

It is important that the sodium acetate should not be added till the liquid has partially cooled, so as to avoid any chance of reaction of the resultant cupric acetate with the glucose in the manner observed by Barfoed.

Pavy's method of determining diabetic sugar by titration with ammoniacal cupric solution would probably be more generally applied if it did not necessitate the use of a special apparatus. To avoid this disadvantage I have devised the following form of the test, which is simple and convenient, but less accurate than where larger quantities of the urine and reagent are employed. An accurately-measured volume of 10 c.c. of Pavy's solution is placed in a wide test-tube, a few fragments of tobacco-pipe dropped in, and 8 to 10 drops of petroleum or paraffin burning oil added. This forms an upper layer which effectually excludes the air. The test-tube is inserted into the neck of a wide-mouthed flask containing hot water, which is then heated until the contents of the tube have reached the point of ebullition. The urine to be tested is treated with an equal measure of ammonia, and filtered from the precipitated phosphates. A known volume of the filtrate is then further diluted with a definite measure of water, according to the proportion of sugar supposed to be present, and then added drop by drop to the boiling hot Pavy's solution by means of a small burette or graduated pipette, until the disappearance of the blue colour indicates the termination of the reaction. If 10 c.c. of Pavy's solution was employed, the volume of urine required to decolorize it contains 0.005 gramme of sugar. Or if 100 grain measures of the copper solution were used, the urine contained 0.05 grain of sugar. By operating in the foregoing manner, fair approximate determinations of sugar in diabetic urine are obtainable very rapidly and with the simplest of apparatus.

Experiments in my laboratory by Mr. G. Bernard Brook show that unclarified healthy human urine exerts a reducing action on Pavy's solution equal to that of a liquid containing from 0.1 to 0.3 per cent. of glucose. G. Stillingfleet Johnson finds the reduction to vary from 0.15 to 0.19 gramme per 100 c.c. (= 0.6 to 0.8 grains per fluid ounce), and ascribes about one-fourth of this to uric acid (removable by lead acetate) and the remainder to creatinine (removable by mercuric chloride).

It is evident, therefore, that Pavy's method, applied in the ordinary manner, is apt to give misleading results when only small quantities of sugar are in question. As to Fehling's test, although, by the foregoing modified mode of application, the indications are much more definite, and the delicacy of the reaction is correspondingly increased, there still remains the disturbance due to the presence of creatinine. On adding Fehling's solution to a solution of this substance, a green liquid is produced, and on boiling a yellow coloration is observed, without, however, any separation of cuprous oxide. It is this behaviour which causes interference with the detection of glucose, the combination of the yellow and blue colours resulting in a green, and in

addition the creatinine compound is said to have the power of preventing the precipitation of cuprous oxide by glucose.

A better separation of creatinine can be effected by a method proposed by Maly and improved by G. Stillingfleet Johnson. The latter chemist adds to the urine 25 per cent. of its volume of a cold saturated solution of mercuric chloride and 5 per cent. of a similar solution of sodium acetate. This reagent effects an immediate precipitation of any albuminous matters which may be present, together with the whole of the phosphates, urates, and most of the colouring matters, and the xanthine bases. If the liquid be filtered immediately, the creatinine remains in solution, but is deposited in the course of a few days. It may, however, be completely thrown down by boiling the liquid for a few minutes. In the filtrate thus purified from all interfering bodies, Mr. Johnson determines the sugar by Pavy's ammoniacal cupric solution, after precipitating the excess of mercury.* Sulphuretted hydrogen is not available for this purpose, since it gives rise to certain fixed derivatives which reduce alkaline cupric solutions. Johnson adds ammonia drop by drop, and thus secures perfect precipitation of the mercury, but this plan has not given good results in my hands, and if the mercury be not completely removed at this stage the subsequent operation is vitiated. I have obtained the best results by boiling the liquid for five or ten minutes with zinc-dust, which precipitates the mercury perfectly. Sixty c.c. of the urine to be tested should be boiled for five minutes with 15 c.c. of saturated mercuric chloride and 3 c.c. of saturated sodium acetate solution, and the liquid filtered hot. The precipitate is washed twice, and the filtrate boiled for ten minutes with zinc-dust and again filtered. The precipitate is washed, and the filtrate diluted to 120 c.c. with ammonia (specific gravity 0.960). This liquid has half the concentration of the original urine, and is added to not more than 50 c.c. of boiling Pavy's solution from a burette in the usual manner. The requisite washing of the mercuric and zinc precipitates can be avoided if 50 c.c. of the urine be boiled with solid mercuric chloride and sodium acetate, the liquid filtered, the filtrate boiled with zinc-dust, and again filtered, and a known volume of the last filtrate mixed with an equal measure of strong ammonia.†

In experiments made in my laboratory by Mr. G. Bernard Brook in the foregoing manner, upon urine from apparently healthy persons, the purified liquid exerted a reducing action on Pavy's solution corresponding to the presence of from 0.05 to 0.13 grammes of glucose per 100 c.c. of the original urine, which yielded crystals of phenyl-glucosazone by the phenyl-hydrazine test. I have proved by parallel experiments on the same sample of urine that Johnson's treatment with ammonia (when successful) and mine with zinc-dust, give the same results. In the case of every urine hitherto examined in my laboratory, there has been a slight residual reduction after the

* Since this paper was read, Sir George Johnson (in an interesting paper published in the *Lancet* for June 7, 1894), proposes to eliminate the creatinine, etc., by boiling with mercuric chloride and sodium acetate, in the manner devised by his son, separate the excess of mercury by ammonia, and determine the sugar colorimetrically in the filtrate by boiling with caustic alkali and picric acid. He finds no brown coloration indicative of glucose to be produced by normal urine which has been previously clarified by mercury.

† This treatment serves the double purpose of keeping the zinc in solution and furnishing a constant but gradually added supply of ammonia during the subsequent titration with Pavy's solution. The additional ammonia has been proved by Mr. C. G. Moor to have no prejudicial effect on the accuracy of the results obtained.

mercury treatment, and I consider it highly probable that these urines, which were limited in number, contained traces of actual sugar. But Mr. Johnson has obtained entirely negative results, which implies that an infinitely large quantity of the urines he examined could be added to Pavy's solution, after purification by mercury, without causing the slightest reduction in the colour.

As all the methods of detecting sugar in urine which are based on the reducing action of glucose are more or less vitiated by the presence of other reducing bodies, a special reagent for glucose has an exceptional value. This exists in phenyl-hydrazine, which, when added as a solution of the hydrochloride to a liquid containing glucose, to which sodium acetate has been also added, gives a yellow precipitate of phenyl-glucosazone. To apply the test, von Jaksch recommends that 50 c.c. of the suspected urine, previously freed from albumin, should be treated with 2 grammes of sodium acetate, and from 1 to 2 grammes of phenyl-hydrazine hydrochloride, and the liquid heated to 100°C. for half an hour; or 10 to 20 drops of phenyl-hydrazine and the same volume of 50 per cent. acetic acid may be employed. On cooling, the phenyl-glucosazone separates as an amorphous or crystalline precipitate of a yellow or brick-red colour. If amorphous, the precipitate should be dissolved in hot alcohol, and the solution diluted with water and boiled to expel the alcohol, when the glucosazone will be obtained in the form of characteristic yellow needles, melting at 205°C., nearly insoluble in cold water, more soluble in hot, moderately soluble in alcohol, and dissolved by glacial acetic acid to form a lævo-rotatory solution. According to von Jaksch, no sugar can be detected by this test in the urine of persons poisoned by arsenic, potash, or sulphuric acid; but the presence of sugar seems constant in the urine of those poisoned by carbonic oxide or other irrespirable gases.*

Instead of operating in the manner prescribed by von Jaksch, the phenyl-hydrazine test may be applied in the following simple manner, which is substantially that recommended by C. Schwartz (*Phar. Zeit.*, xxiii., 465): 10 c.c. measure of the urine is heated to boiling and treated with half its volume, or a sufficiency, of a 10 per cent. solution of neutral lead acetate. The liquid is boiled and filtered hot. Solution of caustic soda, in amount sufficient to redissolve the precipitate which first forms, is added to the filtrate, and then a little (as much as will lie on the point of a pen-knife) phenyl-hydrazine hydrochloride is dropped in. The liquid is then boiled for some minutes, and then strongly acidulated with acetic acid. In presence of much sugar an immediate yellow turbidity or precipitate will be formed, but if only minute traces be present a yellow coloration is first produced, which on cooling and standing changes to a turbidity. In all cases considerable time is required for the complete separation of the glucosazone, but the qualitative indication is readily and quickly obtained.

Unfortunately the phenyl-hydrazine test does not appear susceptible of being applied quantitatively, though, of course, the intensity of the reaction and the amount of precipitate afford a fair indication of the proportion of sugar present.

In all doubtful cases the indications furnished by the production of a turbidity or precipitate with the above test should be confirmed by obtaining the glucosazone in a crystallized form, examining it under the microscope, and, when possible, deter-

* Is it not possible that the traces of sugar apparently present in the urine of some persons in perfect health have their origin in the imperfect combustion of coal-gas?—A. H. A.

mining its melting point. I have found that it is readily dissolved by ether from its acidulated aqueous solutions. On separating and evaporating the ether the glucosazone can be dissolved in alcohol, and crystallized by adding water and evaporating, as already described.

As small a proportion as 0.05 per cent. of sugar can be positively detected in urine by the phenyl-hydrazine reaction. I have obtained distinct indications of sugar by means of it in samples of urine from apparently healthy persons.

Both dextrose and lævulose yield identically the same glucosazone. The only other constituents of urine which simulate the behaviour of glucose with phenyl-hydrazine are glycuronic acid and its compounds.*

Glycuronic acid is a syrupy liquid, miscible with alcohol or water. When the aqueous solution is boiled, evaporated, or even allowed to stand at the ordinary temperature, the acid loses the elements of water and yield the anhydride or lactone ($C_6H_8O_6$), which forms monoclinic tables or needles, having a sweet taste and melting at 167° . It is insoluble in alcohol, but dissolved by water to form a solution which is dextro-rotatory ($[\alpha]_D = +19.25^\circ$), prevents the precipitation of cupric solutions by alkalis, and powerfully reduces hot Fehling's solution, the cupric oxide reducing power being 98.8 compared with glucose as 100.

Glycuronic acid itself is dextro-rotatory ($[\alpha]_D = +35^\circ$), but many of its compounds are lævo-rotatory. It reduces Fehling's solution on heating, and precipitates the metals from hot alkaline solutions of silver, mercury, and bismuth.

With phenyl-hydrazine, glycuronic acid forms a yellow crystalline compound, melting at 114 to $115^\circ C$.

When oxidized with bromine glycuronic acid yields saccharic acid, which can be again reduced to glycuronic acid by treatment with sodium amalgam.

Glycuronic acid is distinguished from glucose by not undergoing the alcoholic fermentation when treated with yeast. On the other hand, when fermented in presence of cheese and chalk it yields lactic and acetic acids.

Glycuronic acid occurs in the urine after the administration of morphine, chloroform, and certain other drugs. In one case recorded by H. H. Ashdown large amounts of glycuronic acid occurred in the urine of a healthy young man, which urine was not abnormal in volume or density.

No method of detecting glycuronic acid has yet been devised short of its actual isolation, and to effect this a large quantity of the urine is required. The method is described in an interesting paper by H. H. Ashdown (*Pharm. Jour.* [3], xx., 607).

DISCUSSION.

The Chairman (Mr. Otto Hehner) invited discussion, and said that Mr. Allen had in the course of his paper afforded another proof of his indomitable industry in the

* Glycuronic acid contains $C_6H_{10}O_7$; or, $COH(CH.OH)_4.COOH$. The substance doubtless has its origin in the dextrose of the body, to which compound it is closely related. It was first obtained in the conjugated form of campho-glycuronic acid, in the urine of dogs to which camphor had been administered, and subsequently as uro-chloralic acid after the administration of chloral. It is remarkable for its tendency to form ethereal or glucosidal compounds when appropriate substances are introduced into the body. Traces of such compounds probably occur normally in urine, especially indoxyl- and skatoxyl-glycuronic acids, in addition to the combination with urea, having probably the constitution of uro-glycuronic acid, which is the ordinary form in which glycuronic acid exists in urine.

collection of facts, and had made an important addition to the literature on the subject of the paper.

Mr. G. Stillingfleet Johnson thought that Mr. Allen's paper was of especial value from a historical point of view. He had gathered together the observations of a number of foreign as well as English chemists on the subject, and had shown how difficult it was to get rid of an error when it once got into chemical literature and text-books. There could be no doubt that Brücke was wrong when he said that sugar was present in considerable quantities in normal human urine, and Bence Jones was wrong in supporting him in this country. But it was probable that the more delicate tests which now prevailed had enabled analysts to overcome these errors, and he thought present-day members of the profession ought not to be too hard upon their predecessors, who were not thus equipped. He thought that a test like the phenylhydrazine test was one of the utmost value. Not only did it apparently produce absolutely no reaction with any normal ingredients of the secretion of the human kidney, but it also appeared to produce no reaction with any of the numerous carbohydrates which were present, probably, in all urines, and which gave reactions with such tests as the alkaline copper test, the bichloride of mercury test, and a number of others. He did not lay very much stress upon Mr. Allen's observation that in some cases he had found sugar in urines, but he laid much more stress on the fact that he had not found sugar by those tests in other normal urines. The question was, What is a normal urine? This was simply reduced to the further question, Who is a healthy man? Everybody knew that a man might rise in the morning a healthy man, and that he might go to bed at night anything but a healthy man. He was quite sure that slight errors of diet—such, for instance, as taking a late dinner, or dining out—were sufficient to produce a temporary glycosuria, which was of no importance whatever. If the test were applied carefully to the urine of healthy individuals, it would be found in the long run to give practically negative results. He had no doubt that the reason why the idea that normal human urines were saccharine in character had existed so long was that the reducing reaction of creatinine had been mistaken for the reducing action of sugar, as Mr. Allen had pointed out. The Germans to this day under-estimated the reducing action of creatinine, which, he did not think, they put at one-eighth of its actual amount, and this he attributed to isomeric changes undergone by the creatinine in the act of isolation from the urine. In isolating this substance from urine, as could be seen by reference to his paper in the Royal Society's Proceedings, vol. xliii., he did not apply heat at a single stage of the process used. All the evaporations were done *in vacuo* over sulphuric acid, and no powerful chemical was used unless it was absolutely necessary; and the result was, as was pretty generally known, that the properties of the creatinine which he had isolated were completely different from those attributed to creatinine by other observers. Its reducing action far exceeded that of any creatinine previously obtained. Now that it was known that creatinine was a powerful reducing agent, and that a test like the phenylhydrazine one, which was capable of reacting with glucose in a human urine, existed, he hoped that the errors which were accidentally introduced so many years ago would at last be rectified, and that a clearer understanding on the matter would in future prevail.

Dr. James Edmunds said that physicians would be grateful to Mr. Allen for reinvestigating this subject of diabetic urines. In life insurance reports and in clinical work there was sometimes a difference of opinion as to how far the reducing action of certain urines depended upon the presence of small quantities of glucose, how far upon the presence of other reducing substances. He trusted that Mr. Allen would show them how to eliminate the non-glycosic reducing substances, and how then to proceed with sure steps to discriminate, and perhaps to quantitatively determine 0.1 per cent., or less, of diabetic sugar.

As to the grass-green coloration shown by Mr. Allen as due to hydrochloride of creatinine, he (Dr. Edmunds) had noted that the blue copper solution also became grass-green under the colorific action of yellow or orange matters in the urine. That this greening was due to colorific action as such, and apart altogether from any chemical reaction on the copper, was proved by simply crossing before the light two tubes, one containing such a urine, and the other containing the dilute Fehling. The same effect was seen by using tubes respectively containing ferric chloride and copper sulphate, each of a suitable depth of colour. And these solutions, if mixed, also gave the grass-green colour.

Another colour fallacy was apt to occur when urine containing sugar was boiled with caustic soda in order to throw out the earthy phosphates, or in titrating back after having added an excess of diabetic urine to the boiling alkaline copper solution. In this way small quantities of sugar might be caramelized, so as to give the filtrate a yellow or brownish tint, such as would at once give a grass-green or bronze-green colour with the Fehling's solution.

Sometimes there came out a muddy precipitate of a terra-cotta colour, which obstinately remained in suspension, until, by reabsorption of oxygen, it went back into a blue solution. This terra-cotta precipitate was probably a hydrated suboxide of copper, kept in suspension, like fine clay, by some urinary substances. But he would like to have Mr. Allen's opinion as to the cause of this reaction.

In hospitals the testing of urines should not be done in the wards. The urines, properly labelled, should be taken down to the pathological laboratory, and tested by a skilled chemist, from whom the students and house-surgeons would learn more than they now did by the objectionable and dangerous habit of keeping urinary reagents in each ward, for the use of students and nurses. He (Dr. Edmunds) once had a patient poisoned in one of his wards by drinking a corrosive acid from a ward-set of urinary reagents.

Mr. Hehner asked Mr. Allen whether he could give any information as to the actual quantity of residual sugar found after treatment with mercuric chloride.

Mr. Cassal wished to know whether there was any likelihood of a sensible loss of sugar by the previous preparation through which the urine passed. It seemed to him that this was very possible.

Mr. Allen, replying to Mr. Hehner, said that Pavy's solution gave perfectly definite and, he believed, true results for the estimation of sugar in urine, if it had been previously put through Mr. Johnson's process. The point on which he differed from Mr. Johnson was that he had not hitherto met with a sample which gave abso-

lutely no reduction by Pavy's test after going through Mr. Johnson's treatment. Although the amount was very small, down to 0.05 per cent., after treatment by Mr. Johnson's mercurial process, he had met with no instance of urine giving a wholly negative result. There was no difficulty in applying Pavy's solution to the estimation of small quantities of sugar, provided that too much of the copper solution was not used. If the results were checked by the production of the crystalline glucosazone by the phenyl-hydrazine reaction, there was a strong presumption of the existence of sugar. Caramelizing of glucose, no doubt, occurred when the Fehling test was applied in the ordinary way. The grass-green colour produced in the cold by creatinine on adding Fehling's solution was very characteristic. Analysts had been in the habit of regarding almost all reductions of Fehling's solution as due to sugar; but, as a matter of fact, there were hundreds of urines which gave a reduction which was not due to the presence of sugar. Whether the residual reduction after treatment with mercury was really due to sugar, as he was inclined to believe, he did not know with absolute certainty. The crystals of glucosazone obtained had been too small in amount in these cases for him to take the melting-point. If glycuronic acid were present, it might have been mistaken for sugar, as it gave a similar crystalline compound with phenyl-hydrazine. He did not quite see why one should be limited to processes involving no heat, in order to isolate creatinine, seeing that it was more convenient to employ heat, the precipitation being then effected in a few minutes, instead of several days. If it were found that creatinine could be extracted from urine in Mr. Johnson's way by means of heat, and that it had when so extracted a constant reducing power, which might be different from that of the substance obtained without heat, a method of estimating creatinine quantitatively could be based thereon. However, he was endeavouring to effect the ready determination of creatinine on another principle.

Mr. Hehner said he quite understood that a determination could be made as to how much urine would be necessary to decolourize a measured quantity of Pavy's solution. But did that afford a measure of the quantity of sugar present? The solution was here used under conditions differing from those prescribed by Dr. Pavy, and he (Mr. Hehner) did not notice that Mr. Allen employed any factor for correction.

Mr. Allen, regarding Mr. Hehner's observation that the introduction of large quantities of saline matters changed the reducing figure for Pavy's solution, said that he had ignored that.*

Mr. Cassal asked Mr. Allen whether there were any effects in the way of loss due to the application of the preparatory process.

Mr. Allen said that he adopted the plan of adding to urine a definite quantity of glucose, and had obtained an increase exactly corresponding to the glucose added.

* Since the paper was read, experiments, made specially with a view of ascertaining the influence of the zinc and sodium salts and the excess of ammonia used, have shown that the error from this cause may be neglected, at least when such small quantities of sugar as those in question are under consideration.—A. H. A.

The Recent Modifications of the Reichert-Meissl Method. C. Bunte. (*Chem. Zeit.*, 1894, xviii., 204-206.)—In defence of his modified Reichert-Meissl method, Kreis has maintained that an acid of definite concentration is essential to success (*ANALYST*, xviii., 166); it must be noted, however, that the specific gravity which he gives for an acid of 91.53 per cent.—viz., 1.825—is not in accord with the value which is assigned by Lunge and Naef to acid of this concentration, viz., 1.8298. Kreis also states (*loc. cit.*) that the correct concentration of the acid is indicated by the rapidity with which it hydrolyses butter-fat and margarine-fat.*

The author has used acids of both the above-quoted and of other specific gravities, and finds that with none of them is there sufficient constancy in the rapidity of hydrolysis to constitute a valid indication of the strength of the acid.

Pinette, Prager and Stern (*ANALYST*, xviii., 145) are correct in their statement that sulphurous acid is always produced during the hydrolysis. The following table gives the mean values obtained in experiments conducted with four acids of different specific gravities and with strict adherence to the prescription of Kreis:

Sp. Gr. of Acid.		Reichert-Meissl No.		Kreis's Reichert-Meissl No.
1.8215	...	27.90 c.c. $\frac{N}{10}$ KOH	...	21.71 c.c. $\frac{N}{10}$ KOH
1.8252	...	27.39 " "	...	25.04 " "
1.8298	...	27.90 " "	...	26.35 " "
1.8393	...	27.90 " "	...	32.33 " "

The duration of the process of hydrolysis was $\frac{1}{4}$ minute with the most concentrated acid, and $1\frac{1}{4}$ minute with the least concentrated, so that the number of c.c. of KOH used is larger the shorter the time of hydrolysis. The sulphurous acid in the distillate increased with the strength of the acid, being equivalent to from 1 to 4 c.c. $\frac{N}{10}$ KOH.

The influence of temperature was next studied, and was found to be very considerable; thus, with the same acid the number obtained was 28.82 c.c. $\frac{N}{10}$ KOH when the fat was "still warm," and 15.55 c.c. KOH when it was at 25° C., at the beginning of the hydrolysis.

It must be concluded that in Kreis's method only a partial hydrolysis, depending for its degree of completion on the strength of the acid and the temperature of the butter-fat is attained, the apparent agreement with the Reichert-Meissl numbers, observed in many cases, being procured by titrating sulphurous acid as volatile fatty acids.

Prager and Stern's suggestions (*ANALYST*, xviii., 145) the author sets aside as impracticable. Pinette (*ANALYST*, xviii., 167) obtains numbers which are too high, either because he heats too strongly or uses too strong an acid; his statement that he adds the acid to the molten fat furnishes no information as to the temperature, because, as is well known, butter-fat melts at 31-31.5° and solidifies at 19-20°.

Inasmuch as the author found that the larger the quantity of butter-fat taken for hydrolysis, the lower the number obtained, it was suspected that the water retained by the fat influenced the action of the sulphuric acid; but in no case was butter-fat found to retain more than 0.21 per cent. of water, a quantity too small to be accused

* In one place Kreis speaks of butter and margarine as being used in this test. Obviously, the varying water-content of these materials would introduce variation in the rapidity of hydrolysis by diluting the acid, and the respective fats must be meant.

of exerting the influence suggested. It is incidentally remarked that filtered butter-fat will give a constant Reichert-Meissl number for many weeks.

The following method is next set forth as the only one which will give constant results, agreeing sufficiently with the Reichert-Meissl numbers: About 5 grammes of the butter-fat are weighed into a litre Erlenmeyer flask, which is then placed in a drying oven and heated to 100° C. Ten c.c. of sulphuric acid (sp. gr. 1·8355) are added, the flask being swung to and fro the while; immersion in a water-bath at 30° to 32° for ten minutes follows, and then 150 c.c. of water are added with constant agitation. The liquid is immediately titrated with a concentrated permanganate solution, until the pink colour is permanent for a few seconds, and further treated as is customary in the Reichert-Meissl method.

Great stress is laid upon the specific gravity of the sulphuric acid, which must agree with that given to at least three places of decimals.

The following table comprises the figures which the author adduces in support of this process.

No.	Reichert-Meissl No.	No. by above Method.	H ₂ SO ₄	Permanganate.
	c.c. $\frac{N}{10}$ KOH	c.c. $\frac{N}{10}$ KOH	Sp. Gr.	
1.	29·44	29·16	1·8355	3·6
2.	29·44	30·68	"	3·6
3.	29·44	30·53		3·6
4.	29·44	30·00		4·0
5.	29·44	29·66		4·0
6.	29·44	29·74		4·0
7.	26·61	26·54		4·0
8.	26·61	27·22		4·0
9.	26·61	27·22		4·0
10.	29·53	29·74		3·6
11.	31·28	29·95		4·0
12.	30·77	30·20		3·9
13.	30·74	29·69		4·0
14.	28·70	28·60		4·0

The results of this investigation may be thus summarized:

(1) The modifications of the Reichert-Meissl method heretofore proposed are either unscientific or impracticable.

(2) The Reichert-Meissl method gives more closely concordant results than the method above prescribed.

(3) The temperature, the concentration of the acid, and the size of the reaction flask must vary between the very narrow limits named, in order that the sulphuric acid method may be at all successful.

A. G. B.

The determination of Carbonic Anhydride in the presence of Soluble Sulphides. A. Wolkowicz. (*Zeit. Angew. Chem.*, 1894, 165; through *Chem. Zeit.*) In the ordinary process of determining carbonic anhydride in the presence of soluble

sulphides CO_2 and H_2S are evolved together, the latter absorbed in a tube of pumice soaked in copper sulphate and the former in a weighed potash bulb apparatus in the usual manner. This plan can be improved by adding cupric chloride to the contents of the evolution flask instead of using a copper sulphate absorption tube, the H_2S being thus retained as CuS , while the CO_2 escapes from the slightly acid solution. The process is particularly applicable for such substances as slag cement.

B. B.

REVIEWS.

THE SALE OF FOOD AND DRUGS ACTS, 1875 AND 1879. By T. C. H. HEDDERWICK, M.A. (Eyre and Spottiswoode. Price 5s.)

This little book, although avowedly written for practising lawyers and justices of the peace, ought immediately to find a place in the library of every public analyst. It contains, in a condensed and most intelligible form, every High Court judgment which affects the working of the Act, and from which the reader can see at a glance how the clauses and verbiage of the Acts have been interpreted by the judges. Of legal quibbles, and of judgments which show ignorance of chemical facts, there are not a few; but on the whole good sound common-sense prevails. Every page contains something of interest to the public analyst, who is bound, if he desire to remain on the list of the "efficients," to know the law as applied to every phase of the Act. Analysts would do well to study carefully the section on the "form of the certificate," and also the complications of the warranty question. We specially commend to their attention the admirable judgment of the late Lord Chief Justice Coleridge, who said: "This Act was passed with the object, not of punishing the seller, but of protecting the buyer, and of insuring, as far as it is possible to ensure, such a result that a person who buys an article of a particular description should get a genuine article, and one of which *contains the proper quantity of the different elements that an article of that description ought to contain.*" After such a decision, what becomes of the milks from starveling cows; of limits designed to pass exceptionally poor milks as genuine standards; and of the absurd contention emanating from Somerset House, that as long as an article has had nothing added to it, or abstracted from it, the law holds it to be genuine?

It is a pity that the little book does not also deal with the Margarine Act, the decisions concerning which can hardly be separated from those given under the Food Acts.

Appended to the book are notes on "Milk Adulteration," consisting of extracts from the evidence given before the Parliamentary Committee of 1872, mainly by the late Drs. Tidy and Voelcker. These tended to show that, in their opinion, genuine milk may contain as little as 10 per cent. of total solids, and that no milk with more than 10 per cent. ought to be condemned on analysis alone. Though the basis upon which these conclusions were founded has long been shown to be a fallacious

one, yet analysts would do well to make themselves thoroughly acquainted with the facts, or alleged facts, because after this book has come into the hands of defence solicitors, as it no doubt widely will, we shall hear a great deal more of the product of these wretched starveling cows and other "animated bundles of bones." O. H.

NATURE'S HYGIENE. By C. T. KINGZETT, F.I.C. London: Baillière, Tindall and Cox.

That a fourth edition of this book has been called for since its first publication in 1880 is a matter of congratulation to the author, and a proof that it has met with general acceptance by the class of readers for whom it was intended. It is especially suitable for those who have little or no knowledge of chemistry, and it gives a clear summary of each of the separate subjects which it embraces. These, as will be seen from a glance at the chapter headings, are far too wide to be more than lightly touched upon in the short space of five hundred pages. At the same time, the author has found room to notice many of the latest discoveries.

As in previous editions, the book is divided into two parts. The first, after an account of the general principles of chemistry, and especially of oxidation, deals somewhat more at length with sanitary subjects, such as water, sewage, bacteria, and disinfectants. In the second part, the various industries connected with the pine and eucalyptus are described, and an account is given of experiments made by the author to prove the disinfecting properties of oxidized terpenes.

The chapter on sewage is perhaps one of the best, and no clearer digest of the different methods of treatment could be desired. In this connection it may be mentioned that the statement on page 83, as to the probability of pathogenic organisms in sewer gas, is not altogether in agreement with recent experiments. In a report* on the subject to the London County Council Mr. Laws stated that he had found that the organisms in the gas issuing from sewers were in almost every case non-pathogenic, and that the two micro-organisms, *bacillus coli communis* and *micrococcus ureæ*—both of which must be present in abundance in sewage—were conspicuously absent from the gas. The immunity which sewer men enjoy from zymotic disease seems to point in the same direction, as does also the work carried out by Messrs. Carnely and Haldance on the sewers of Westminster Palace and of Dundee.

In the section dealing with bacteria and their products the graft theory of disease is described at some length, but little mention is made of the at least as probable theory that the protozoa are the cause of certain diseases (*e.g.*, cancer) of which the bacterial origin cannot be proved. A short description of the different diseases and the micro-organisms associated with them is given in pp. 283-306; but the frequent mention of a well-known excellent disinfectant might have been spared in a work which professes to be of a purely scientific character. With the remarks of the author in the preface on the importance of having properly qualified chemists to do the work which is now often done by the incompetent, we cordially agree.

C. A. M.

* Report to the Main Drainage Committee of the London County Council on Sewer Air Investigation, by J. PARRY LAWS, F.I.C.

MICRO-ORGANISMS IN WATER: THEIR SIGNIFICANCE, IDENTIFICATION AND REMOVAL.
By PERCY FRANKLAND, Ph.D., B.Sc. (Lond.), F.R.S., and Mrs. PERCY FRANKLAND. London: Longmans, Green and Co. Price 16s. (net).

The bacteriological investigation of water has now assumed a highly-important place besides that of the usual chemical analysis, and as time progresses this is certain to become more and more the case. It is highly desirable, therefore, that every analyst engaged in the examination of drinking-water should possess an intimate acquaintance with the biological side of the question; and to those who are not *au fait* in this direction, the work before us will be found of the greatest possible assistance, whilst its appearance will be hailed with satisfaction by all earnest workers in this department. The names of the authors are in themselves a guarantee as to the ability and thoroughness which would be brought to bear upon any task of this nature which they might undertake, and it must be freely admitted that in this work they have fully maintained their reputation. In addition to the record of an enormous amount of original work, almost everything of importance which has appeared in English or foreign literature in relation to the question is referred to, and its source duly acknowledged; indeed, the reader cannot fail to be struck with the immense amount of literature which the authors have perused in the course of their investigations during the last ten years.

The work is divided into nine chapters and an appendix. The first two chapters treat on the operative details of biological work; and whilst these are given in a terse form all that is necessary is included, and the directions are stated in such an intelligible manner that no one should find the slightest difficulty in following them. Chapter III. describes the actual examination of water for micro-organisms, and contains important information as to the collection of samples, the general and particular appearance of colonies, etc. The next chapter, which is a very important one, treats on the purification of drinking-water and of sewage effluents by the various methods of filtration commonly in use. It not only shows the importance of bacteriological examinations in determining the amount of work which each individual construction of filter is capable of accomplishing, but also in ascertaining afterwards whether such filter is performing its work efficiently or otherwise. Quite a new light is thrown upon the beneficial effects of filtration; it is shown that these are not so much due to an alteration in the chemical constitution of the water as to the amount of micro-organisms eliminated. This is proved by a very extensive series of experiments undertaken by one of the authors, the results of which are both extremely interesting and surprising. Chapter VI. treats of the multiplication of the organisms in water under varying conditions; Chapter VII. on the detection of pathogenic species, and gives many remarkably ingenious processes which have been contrived to obviate the, in some cases, almost insuperable difficulties which attend their identification. Chapter IX. is devoted to a description of the action of light on bacteria; and lastly comes the Appendix, which is one of the most important divisions of the whole book; it contains a list, numbering upwards of two hundred, of all the micro-organisms which have been so far found in water, their microscopic appearance, their behaviour when cultivated in or on the several mediums, together with other details necessary for their identification.

The work is printed in bold, clear type, illustrated with engravings where necessary, neatly bound, and is provided with a good index. We can recommend it with every confidence as a work unique in character and absolutely indispensable to all who are engaged in the sanitary examination of water.

W. J. S.

THE ANALYST.

SEPTEMBER, 1894.

THE LATE C. R. ALDER WRIGHT, B.Sc. (VICT.), D.Sc. (LOND.), F.R.S.

It is with feelings of the deepest regret that we have to record the somewhat sudden decease, at the comparatively early age of forty-nine years, of an old and much esteemed member of the Council of the Society of Public Analysts, Dr. Alder Wright, which took place on July 23.

In his early life it was intended that Dr. Wright should follow the profession of an engineer, and for this object he received a special training; but, feeling a strong attraction towards chemistry, he eventually forsook the former pursuit, and devoted his attention to the latter, in which he was soon destined to make his mark.

A survey of Dr. Wright's professional career shows it to have been one of exceptional activity. In 1866, whilst yet a student at Owens College, Manchester, he contributed a paper to the Chemical Society on the "Action of Light on Sensitive Photographic Papers." Having completed his college career, he became chemist to the Runcorn Soap and Alkali Works, and in 1867 read a valuable paper before the Chemical Society describing his experiences in this capacity. He next became assistant in the laboratory of St. Thomas's Hospital, and about this time contributed a number of letters to the *Chemical News* on the various methods of testing alkalies, and on other subjects of interest from a technical point of view. He also published the results of a large number of investigations on bodies of the most diverse nature, some of which he had made independently, others in conjunction with Matthieson and others. Amongst these are included his valuable researches on the alkaloids of opium and of the aconite family. As exhibiting the mathematical bent of Dr. Wright's mind may be adduced the series of nine voluminous papers, which appeared for the most part in the *Philosophical Magazine*, "On the Determination of Chemical Affinity in terms of Electromotive Force." In 1873 he contributed a paper to the Chemical Society on "The Hydrogen occluded by Palladium," and, later on in the same year, a series of papers on "Isomeric Terpenes." In 1875 Dr. Wright delivered a lecture at the Royal Institution on "The Chemical Changes accompanying the Smelting of Iron in the Blast-furnace," which bears evidence of the large experience he had gained in the research laboratory and works of Sir Lowthian Bell. Then follows a series of papers, commencing in 1878, which were communicated to the Chemical Society on "Researches on some Points in Chemical Dynamics," followed by a paper in 1880 on "Notes on Manganese Dioxide." He was elected a fellow of the Royal Society in 1881, to which he communicated papers in 1889 and 1892 on "Ternary Alloys." In 1884 Dr. Wright read a paper before the Society of Arts on "Cupro-ammonium Solutions, and their use in Waterproofing Paper and Vegetable Tissues," and in the following year gave a course of three Cantor Lectures on the "Manufacture of Toilet

Soaps." Amongst his published works are a treatise on "Metals, and their Industrial Applications," which appeared in 1878, and the one on "Animal and Vegetable Fixed Oils, etc.," which appeared quite recently.

THE QUANTITATIVE SEPARATION AND ESTIMATION OF THE ALKALOIDS OF COCOA.

BY WILLIAM E. KUNZE.*

THE term "alkaloids," according to the most recent views on the constitution of these bodies, should be reserved for those substances of a basic nature which are found ready-formed in plants, and which are constitutionally derivatives of pyridine. Since theobromine and caffeine are xanthine derivatives, they entirely differ in chemical constitution from the alkaloids proper. They should, consequently, be separated from these and included with the nucleins, which form a subdivision of the proteids. It is a matter of some difficulty to find a generic name for these two bodies which, whilst being precise, clear, and not too cumbersome (cocoa-xanthines?), shall sufficiently define their isolated position and sharply distinguish them from the alkaloids. For this reason the author has retained their old designation (alkaloïde) in the present paper. The variety of the methods which have been proposed from time to time for the estimation of these bodies, their number, and the fact that every fresh investigator has propounded a new process, affords distinct evidence that there has not existed, up to the present time, a thoroughly reliable method for their determination. The following is a short *resumé* of the methods which have been hitherto proposed :

1. *Weigmann's Method*.—20 grammes of cocoa are rubbed up with hot water, boiled from a quarter to half an hour, made up to 1 litre, and allowed to settle. 500 c.c. of the clear liquid are precipitated, boiling, with ferric acetate, filtered, the filtrate concentrated by evaporation, 6 per cent. of its weight of H_2SO_4 added and precipitated with phosphotungstic acid. The precipitate is washed with dilute H_2SO_4 , and the amount of nitrogen which it contains estimated; from this the quantity of theobromine deduced.

2. *Mulder's Method*.—10 grammes of cocoa are rubbed up with water, boiled for a quarter of an hour with magnesia, and evaporated to dryness, the dried mass exhausted with chloroform, and the latter distilled off. The residue from the chloroform is dissolved in hot water, filtered, evaporated to dryness, the residue remaining weighed, ignited, and again weighed. The difference between the two weighings gives the quantity of theobromine. The chloroform extract also contains the theine. For its separation from the theobromine Weigmann suggests the following modification: the mixture of cocoa and magnesia is first extracted with benzol (in which theobromine is almost insoluble), the solution evaporated to dryness, the residue shaken out with hot water, the filtrate evaporated to dryness, and the residue weighed as theine.

(Schmidt-Pressler has also pointed out the difference in solubility of the two

* Abridged from the original, which appeared in *Fresenius's Zeit. f. Analyt. Chem.*, 1894, p. 1.

alkaloids in benzol, and called attention to the possibility of their separation in this way.)

3. *Wolfram's Method*.—10 grammes of cocoa, or 20 to 30 of chocolate, are treated for a considerable time with boiling water, ammoniacal lead acetate is then added in slight excess, the mixture filtered hot, and the precipitate washed with hot water until the acidified filtrate, after cooling, no longer gives a precipitate with sodium phosphotungstate.* To the filtrate, which, when excess of lead acetate has been employed, is perfectly brilliant, a solution of caustic soda is added, the whole evaporated to 50 c.c., strongly acidified with H_2SO_4 , and filtered. The theobromine is separated out of the filtrate by the addition of sodium phosphotungstate in considerable excess, with stirring and gentle warming, allowed to become cold, the precipitate filtered off, and washed with a 6 to 8 per cent. H_2SO_4 solution. The washed residue is decomposed by warm barium hydrate solution, the excess of baryta removed with H_2SO_4 , the filtrate neutralized with barium carbonate, filtered hot, and the residue washed with hot water. The united filtrate and washings are evaporated in a platinum dish, dried and weighed. The residue is ignited, the ash treated with ammonium carbonate, the liquid evaporated, and the residue dried, and weighed. The difference between the two weighings gives the quantity of theobromine.

4. *Legler's Method*.—This is essentially a modification of Wolfram's. 20 to 25 grammes of cocoa, or 50 grammes of chocolate, are, after removal of the fat, digested on the water-bath for some hours with 50 c.c. of 4 to 5 per cent. H_2SO_4 , the solution filtered, and the theobromine precipitated by sodium phosphotungstate in excess. The precipitate, which is contaminated with proteids, is allowed to settle for twenty-four hours and filtered off, washed with 8 to 9½ per cent. H_2SO_4 , and dissolved in sodium carbonate solution. The slightly alkaline solution is evaporated to dryness, quartz sand being added, and the residue extracted with amyl alcohol on the water-bath at a temperature of 80° to 90° C.; the solution evaporated in a platinum dish, and the residue dried and weighed. It is then gently ignited and again weighed, the difference giving the theobromine.

5. *Trojanowski's Method*.—A weighed quantity of cocoa in powder is extracted with petroleum spirit to remove fat, well rubbed up with powdered glass and water, mixed with a weight of magnesia equal to that of the cocoa, and dried at a temperature of 60° to 70° C. The dry mass is sprinkled with alcohol and rubbed to a fine powder, boiled with 50 c.c. 80 per cent. alcohol under a reflux condenser for half an hour, this operation being again repeated with another 30 c.c. alcohol for a quarter of an hour. The extracts are united and filtered boiling hot into a beaker, and evaporated on the water-bath; the residue is brought on to a filter with petroleum spirit and washed with alcohol, until the washings run off nearly colourless. The residue on the filter is then weighed, and $\frac{1}{11.75}$ of the weight of the alcohol used in washing added as a correction of the solubility of the alkaloid in this fluid.

6. *Zipperer's Method*.—The beans, from which the fat has been previously removed by petroleum spirit, are extracted three times with 80 per cent. alcohol, the extracts united, calcium hydrate added, and the whole evaporated to dryness on

* The sodium phosphotungstate is prepared by dissolving 100 grammes of sodium tungstate and 60 to 80 grammes of sodium phosphate in 500 c.c. of water acidified with nitric acid.

the water-bath. The dry residue is extracted with boiling chloroform in a Soxhlet apparatus. The chloroform extract is evaporated to dryness, and the residue dissolved in boiling water, filtered, and evaporated to dryness in a platinum dish, dried and weighed; 0.89 per cent., representing water, is deducted from the weight found.

7. *Süss's Method*.—6 grammes of an intimate mixture of equal parts of cocoa and quartz sand are extracted for ten hours with petroleum spirit, boiled for half an hour with 200 c.c. distilled water and 6 grammes of freshly prepared pure lead oxide, strained, the residue squeezed, and the fluid thus obtained filtered. The cocoa residue is again twice boiled, each time with 100 c.c. distilled water for a quarter of an hour, and similarly treated. The nearly colourless filtrate is evaporated down to 10 c.c. and shaken out three times with chloroform, 100 c.c. being used each time (time of shaking, three minutes). The bulk of the chloroform removed by distillation, and the remainder evaporated to dryness in a weighed glass vessel.

8. *Diesing's Method*.—The chocolate, after previous removal of the fat by petroleum spirit, is extracted with chloroform for four hours in a Soxhlet, the residue from the evaporation of the chloroform boiled with water several times, and to these solutions the water is added in which the fat had been boiled. The united fluids are then evaporated to dryness in a platinum dish and the residue weighed as pure theobromine.

9. *Bell's Method*.—20 grammes of cocoa are repeatedly digested with benzol, filtered, and the united filtrates distilled; the fatty residue boiled with water, allowed to cool, and filtered. This process is repeated twice, so that the alkaloid may be completely extracted by the benzol. The aqueous extract is then evaporated to dryness, and the residue purified first by solution in water, then in benzol. In this way an alkaloid resembling theine is obtained in white silky crystals. The cocoa, after the treatment with benzol, is rubbed up to a magma with water, sand, and 10 grammes of calcined magnesia added, and the mixture, after being dried in the water-bath, repeatedly treated with alcohol. After removing the alcohol by distillation, the residue containing the theobromine is brought into a glass, dried, and weighed; the last traces of fat and of the other alkaloid removed by treatment with hot benzol, the latter being separated (?) and weighed. The residue insoluble in benzol is twice treated with a little ice-cold water, and in this way the theobromine obtained by decantation is a perfectly pure and white powder, containing a trace of inorganic matter. The amount of the latter is determined by incineration. As even cold water in some cases (?) dissolves a portion of the theobromine, it is advisable to evaporate the aqueous extract to dryness, and if a small quantity of crystals are found, to treat it with water once more (?). "If the whole residue, obtained by extracting the cocoa and magnesia mixture with alcohol, be submitted to a nitrogen estimation, and the nitrogen found calculated to theobromine, a greater and probably more exact percentage amount of the alkaloid would be obtained."

Criticism of the above Methods.—In Legler's method (No. 4) no directions are given as to the nature of the solvent to be used for the extraction of the fat, and this is by no means a matter of indifference, since the alkaloids are soluble in some of these; and, moreover, as Diesing has shown, the solubility of cocoa-fat in the

different solvents varies. Diesing distinctly prefers petroleum spirit to ether or chloroform. Amylic alcohol is an extremely unpleasant liquid to use on account of its physiological effects.

Trojanowski's method (No. 5) is faulty, owing to the necessity of having to make a correction for the solubility of theobromine in the wash-liquid. Moreover, Trojanowski has not used a correct solution-factor, the solubility of theobromine, according to other observers, being only about a tenth of that which he gives, namely, 1 : 12,500 or 16,600. From the author's experiments it is 1 : 7,100 (absolute alcohol at 20° C.).

Zipperer's method (No. 6) is not clearly described; there is no exact statement of the quantities to be used; and there are also other obscurities, to which must be ascribed the low results which he obtained in comparison with those of the author.

Süss evidently came to the conclusion by comparative experiments that the methods of Zipperer and Diesing gave results below the truth. He perceived that the method they employed (direct extraction of the alkaloid with chloroform) was the cause of this; he therefore altered the method of procedure to that of shaking out the alkaloid from its aqueous solution. In this way he obtained nearly twice as much alkaloid as Zipperer and one and a half times as much as Diesing.

After Bell (No. 9) had demonstrated the presence of another alkaloid in cocoa besides theobromine, and Trojanowski had also called attention to the presence of some other body, of which, from lack of sufficient material, he was unable to ascertain the real nature, and after Schmidt had fully identified it with caffeine, it became apparent that the majority of workers had entirely overlooked its presence, and contented themselves with the estimation of the theobromine alone (Mulder, Wolfram). Zipperer expressly insists on the removal by petroleum spirit of the caffeine along with the fat, and his results, therefore, represent pure theobromine, that is, providing the whole of the caffeine is removed in this way. He, however, like the others, entirely neglects to estimate the caffeine. Diesing has remarked that in the extraction of the cocoa-fat a part of the caffeine is also removed; he therefore directs the fat to be boiled in water, and this solution added to that containing the bulk of the alkaloid. He overlooks the fact that it is caffeine which goes over with the fat, and erroneously calls his residue pure theobromine. Since both caffeine and theobromine are precipitated by sodium phosphotungstate, and as they contain different amounts of nitrogen, Weigmann's method must lead to incorrect results. In the methods of Legler, Süss, and Trojanowski the caffeine passes either partially or completely into the fat solution, and escapes detection. The objection to Mulder's method, as stated above, is that the alkaloid obtained is regarded as pure theobromine. This must be taken with a limitation, since, according to the length of time of the extraction with petroleum spirit, the caffeine may be wholly or only partially extracted. According to this, the correctness of the methods of Süss, Trojanowski, Zipperer, and Legler hinges on the fact as to whether the caffeine is partially or totally removed by the petroleum spirit. In the methods of Mulder, Wolfram, and Diesing the results are not reckoned upon pure theobromine, but on a mixture of the two alkaloids. Weigmann's method must be totally rejected. The method of Bell, and Weigmann's modification of Mulder's, possess points of importance.

The author gives the following results, which he has obtained from the same sample of cocoa examined by the different methods :

Method.	Grammes Found.				Expressed in per cents.			Remarks.
	Grammes of Cocoa taken.	Total Alkaloids.	Theobromine.	Caffeine.	Total Alkaloids.	Theobromine.	Caffeine.	
1. Weigmann ...	10	—	0.0652	—	—	0.652	—	N found = 0.0203 grammes, reckoned as theobromine.
2. Mulder mod. by Weigmann	10	0.059	0.0475	0.0115	0.59	0.475	0.115	Estimated by difference.
3. Wolfram ...	10	—	0.215	—	—	2.15	—	Ditto.
4. Legler ...	20	—	0.0785	—	—	0.3925	—	Ditto.
5. Trojanowski ...	10	—	0.564	—	—	5.64 (!!)	—	Coloured deeply brown, greasy, not decolorizable by washing with alcohol.
6. Diesing ...	10	—	0.124	—	—	1.24	—	Coloured, carbonized on heating.
7. Zipperer ...	10	—	0.0235	—	—	0.235	—	Not absolutely colourless.
8. Süss ...	3	—	0.054	—	—	1.80	—	Slightly coloured. A little ash.

The differences in these results are striking, varying, as they do, from 0.235 per cent. (Zipperer) to 5.64 per cent. (Trojanowski). In the determinations by the methods of Zipperer and Legler the theobromine has evidently escaped the solvent action of the alcohol. The author's results obtained by the methods of Zipperer, Diesing, and Süss agree closely with those obtained by Süss, who obtained from 3 grammes of cocoa the following amounts :

Zipperer	0.7 to 0.8%	Theobromine.
Diesing	1.0 „ 1.1	„
Süss	1.4 „ 1.46	„

The analysis by Trojanowski's method gives most improbable results.

The author considers that in any rational method of analysis of the cocoa alkaloids the total quantity of the two together should be first ascertained, one of the bases should then be isolated from this. If, during the fat extraction with petroleum spirit, a partial solution of the theobromine is not to be feared, there yet remains the possibility of small traces being carried over mechanically. The principal reason, which demands that the total amount of the two alkaloids should be first ascertained, lies in the difficulty of removing the caffeine without losing some traces of the theobromine ; and, besides this, there is no criterion to judge when the whole of the caffeine is removed during the fat extraction. The common fault of all the

methods hitherto proposed is that they entirely neglect the estimation of the caffeine, which is a body of no less importance in its physiological effects than theobromine. It has become the rule to estimate the value of such food substances as tea, coffee, cocoa, etc., by the amount of alkaloids which they contain; possibly also the relative proportions of the two alkaloids in a cocoa might give some clue to its origin.

With reference to the influence of cocoa-fat on the estimation, the author has proved that petroleum spirit, either pure or holding cocoa-fat in solution, dissolves caffeine in considerable quantity; Süss has shown that theobromine is absolutely insoluble in these fluids, but he found that small quantities of this body were carried over mechanically in the fat extraction. The author found that the cocoa alkaloids could be readily extracted by a solution of the caustic alkalies with formation of the respective salts, and that the bases were reprecipitated on the alkali being converted into carbonate. He thought that in this way there might be founded a satisfactory process for the estimation of the alkaloids, but the concomitant saponification of the fat proved an insurmountable obstacle. Evidently for a process of this nature to succeed, the fat must be first removed; for this purpose various solvents were tried: benzol, toluol, xylol, chloroform, methyl alcohol, benzyl alcohol, etc., none of which proved suitable, since they all dissolved more or less caffeine or theobromine. The attempt to devise any process which required the previous extraction of the fat was abandoned.

Though the two methods—extraction and precipitation—are equally correct in principle, the latter method is to be preferred in practice, because no solvent is known which will dissolve the alkaloids without simultaneously taking up other substances, and also because with the small quantities of material usually employed the necessary purification of the alkaloid, either by re-crystallization or taking up in another solvent (shaking out), is certain to lead to loss of material unless the work is carried out with the greatest possible care.

The method of determination may be either: 1. Direct weighing; 2. By difference (loss on ignition); 3. Weighing a definite (metallic) compound; 4. By titration.

No. 1 presupposes the purity of the residue; by No. 2 any non-volatile constituent can be deducted; the platinum compounds alone fulfil the conditions of constancy required by No. 3, and permit of a ready control by igniting, and weighing the residue of platinum. The general method of titration proposed by Barth, which depends on the dissimilar reactions of the alkaloids with litmus and phenolphthalein, is inapplicable in this case, since theobromine does not behave towards these indicators in the same way as the other alkaloids. The greater number of the proposed methods prescribe direct weighing; two determine by loss on ignition, one by a nitrogen estimation. Direct weighing possesses the advantage that one has the actual body in hand, which can, when necessary, be identified and tested for purity; but the before-mentioned difficulties in the way of obtaining the alkaloid free from foreign admixtures, and the loss likely to ensue during its purification, considerably detract from its value.

In testing a method for correctness, it is no satisfactory evidence to show that it yields higher results than previous methods; it must rather be demonstrated that the

method is absolutely quantitative and includes no sources of loss or error. This can only be proved by test experiments. In the present case, on precipitating a known quantity of the alkaloid with phosphotungstic acid, regeneration of the alkaloid, etc., the quantity found must exactly correspond with the amount taken. When it has been found that this is so, an investigation must be made to discover if the other bodies present exercise a disturbing influence on the process. In order to determine this, a second control experiment must be made, which shall represent as completely as possible the actual conditions of working. With this object in view the author has made parallel determinations in which a known mixture of the two alkaloids was employed. The difference in the two determinations must, if the process is to be credited with success, exactly represent the quantity of the added material.

From the above considerations a rational process for the estimation of the cocoa alkaloids ought to be founded on the following principles:

I. The estimation, which may be divided into two phases:

1. Determination of the total alkaloids collectively.
2. Their quantitative separation.

II. Previous extraction of the fat is not permissible.

III. The total amount of the alkaloids must be estimated by weight.

IV. The method of estimation must be controlled for exactitude in its various stages and in its whole course by parallel experiments.

V. The alkaloids must be separated in such a form as will permit of their subsequent identification.

In the author's method water is employed as the solvent, since alcoholic fluids and chloroform are excluded on account of the subsequent difficulties in purification, and in order that the fatty and alkaloidal solutions may be kept quite separate. Acid and alkaline solutions were inadmissible, the former because the conversion of the alkaloids into their respective salts might hinder the transference to another solvent, the latter on account of saponification difficulties. The author intended, first of all, to bring the alkaloids into solution with boiling water, then to evaporate the solution to dryness with the addition of a binding material to retain the disturbing admixtures (colouring matters, sugar, etc.), and then exhaust the residue with a suitable solvent. Taking into account the conversion of caffeine into caffeidine, which ensues on long boiling with caustic bases (*e.g.*, $\text{Ba}(\text{HO})_2$), and the decomposition of theobromine on being evaporated with CaO (Diesing), the author made choice of MgO on account of its lesser basicity. Süss has controverted this statement of Diesing's, and to all appearance correctly, for the author found the destruction of the theobromine only partial; he points out that in an estimation conducted according to Zipperer's method the small quantity of theobromine obtained from the residue which contained calcium hydrate points to its partial decomposition, or to the formation of an insoluble calcium compound. He considers that chloroform is to be preferred to amyl alcohol as the solvent. An experiment in which a weighed quantity of theobromine was evaporated to dryness with MgO and extracted with chloroform in a Soxhlet, showed considerable loss, probably owing to the formation of a magnesium compound. On account of the solubility of MgO in boiling chloroform, this fluid could not be used in this condition. Hence the author was obliged to abandon this method and attempt

the isolation of the alkaloids by shaking out with chloroform as proposed by Süss. This necessitated the removal of the colouring matters and other impurities which partially dissolve in that fluid. Treatment with PbO, animal charcoal, or basic ferric acetate, [did not suffice to completely decolorize the aqueous solution. The author succeeded in effecting this object by the addition of animal charcoal and aluminium acetate, which, upon prolonged boiling, removed every trace of colour; but traces of tannin and considerable quantities of sugar still remained in solution. The filtrate was then evaporated to 10 c.c., and shaken out several times, each time with an abundance of chloroform; on subsequent evaporation of the united extracts, the residue was not, however, obtained in the requisite state of purity, a brown greasy substance obstinately clinging to it. Probably some reaction took place between the aluminium and the theobromine, for in a control experiment the alkaloid could not be recovered quantitatively. As Mitscherlich has stated, theobromine is retained by animal charcoal; this was proved by direct experiment; of the 0.0375 gramme taken only 0.0057 was recovered. In this method of determination it is assumed that the alkaloids are contained in the cocoa in a form soluble in water; should they be combined with tannin or with organic bases or acids, then it is questionable if they can be extracted by water alone. The precipitation methods offer the essential advantage that an acid medium, which has a more powerful solvent action, may be employed for extraction; the presence of an acid also facilitates the subsequent precipitation. The best reagents for the precipitation are the double phosphates, sodium phospho-tungstate, -molybdate, -antimoniate. The precipitate thus obtained can be decomposed by an alkali, when it yields either the corresponding salt of the alkali, or the free alkaloid. When this is effected by NaHO a clear solution results; on passing CO₂ through this the alkaloid is liberated, and a portion, beyond that which is soluble in the medium, is precipitated. The alkaloid may be also liberated by adding to the precipitate a magma of BaCO₃. The precipitation was complete, for in the neutralized residue, obtained by evaporating the filtrate, no trace of alkaloid could be detected by the murexide reaction (chlorine water and ammonia). In a control experiment where a known weight of theobromine was dissolved in about 5% H₂SO₄, the solution treated with phosphomolybdic acid, the precipitate dissolved in NaHO solution, this treated with CO₂, and the residue obtained on evaporation boiled with chloroform under a reflux condenser, the alkaloid was recovered as a pure white powder, containing a minimum amount of ash, thus:

Theobromine taken	0.0605 gramme.
Residue	0.0608 "
Ash	0.0003 "
Theobromine found	0.0605 "

In a second trial 0.053 gramme theobromine were taken, and 0.054 gramme theobromine + ash recovered. When the decomposition is effected by baryta water similarly satisfactory results are obtained. It is impossible to obtain the alkaloid absolutely ash-free, consequently in very exact determinations a weighed portion must be ignited and the ash deducted, not forgetting its re-carbonation with ammonium carbonate.

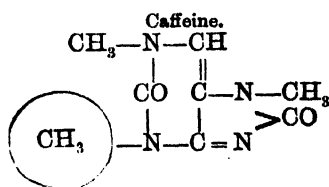
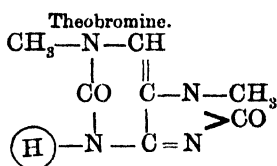
Phosphomolybdic acid was found to be the best precipitant, since the precipitate yielded is of a flocculent nature, and subsides more readily than those obtained by the other reagents. When NaHO is used to liberate the alkaloid, the extraction with chloroform is effected more easily than when $B.(HO)_2$ is employed. In the latter case the ash is lower, and is in such minimum quantity that it may be safely neglected. As the decomposition of the precipitate takes place momentarily, the author considers that no action on the caffeine is to be feared. To test the reliability of the process when other substances were present, the author made the following control experiments. The alkaloids from 10 grammes of cocoa were estimated, a parallel experiment being made with the same cocoa to which a known weight of a nearly equal mixture of the two alkaloids was added. The following are the results obtained :

10 grammes of cocoa yielded	0.1195 gramme alkaloids.
Ditto + 0.107 gramme alkaloids	0.2255 " "
Added	0.107 " "
Difference found	0.106 " "

The exact details of the method are as follows: 10 grammes of cocoa are boiled for twenty minutes with about 150 c.c. of 5% H_2SO_4 , filtered, and the residue thoroughly washed with boiling water. Excess of phosphomolybdic acid is added, and the whole kept warm for twenty-four hours, filtered off, and washed with approximately 5% H_2SO_4 (800 to 1,000 c.c.). The filter and precipitate are brought, still moist, into a beaker, baryta water added, and CO_2 passed through until the whole of the barium is precipitated. The whole is then evaporated to dryness, dried, the residue carefully removed to a flask, in which it is exhausted with boiling chloroform under a reflux condenser. The chloroform solution is filtered into a small Soxhlet glass, such as is used in fat extractions, the fluid distilled off, and the residue weighed. The mixture of alkaloids thus obtained is invariably pure white, almost ash-free, and gives the murexide reaction. Zipperer found a sticky organic substance which clung persistently to the theobromine obtained by alcohol and chloroform extractions. The author has not found this to occur in quantity sufficient to affect the accuracy of the analysis. The solution of the alkaloidal residue in ammonia is slightly opalescent, foams on boiling, and the theobromine obtained on separation (see further on) was not absolutely white. This may be caused by the body described by Zipperer, or by some other. It does not, however, interfere with the estimation, or with the subsequent separation of the two alkaloids, nor was there observed in the subsequent treatment with ammoniacal $AgNO_3$ any blackening indicative of reduction, nor was the recovery of the theobromine from the silver salt in any way interfered with by it. The coloured compound of silver and theobromine dissolves in dilute HNO_3 to a nearly colourless solution; by subsequent neutralization, evaporation, and extraction with chloroform pure theobromine is obtained in beautiful crystals.

Having now devised a reliable process for the estimation of the total amount of the two alkaloids, the next point was to find a similarly reliable one for their separation. The simplest would be a physical method, depending on the solubility of one and the insolubility of the other in some fluid such as benzol, petroleum

spirit, etc. Experiments were made with these and many other liquids, but as no satisfactory results could be obtained in this way, the method was abandoned. The attempt to find a reagent which, while precipitating the one, should leave the other intact, failed, it being found that both behaved in a similar manner with the ordinary alkaloidal reagents. Phospho-antimonic, -tungstic, -molybdic acids, and bismuth-potassium iodide (more energetically with theobromine than with caffeine), precipitated both bodies. Iodine and potassium iodide, and gallic acid precipitated neither. The following reagents were also without effect on either alkaloid: alcoholic ammonium sulphide, iodic acid, iodine trichloride, ferrous and ferric chlorides, Millon's reagent, potassium xanthate, basic and neutral lead acetates, chromic acid mixture in the cold, disodium phosphate, potassium ferrocyanide, sodium carbonate, copper sulphate, basic copper acetate. In solutions of theobromine, *but not of caffeine*, mercuric chloride produced a turbidity which formed very slowly; picric acid in HCl solution yielded a gradual crystalline separation; ammoniacal AgNO_3 solution gave, on prolonged boiling, a crystalline precipitate. A solution of *either* alkaloid, when treated with sulpho-molybdic acid, became, on boiling, of a dark-green colour, which spontaneously changed to indigo-blue. Urea and uric acid also gave this last reaction, which may, therefore, be regarded as common to the whole group. The most powerful alkaloidal precipitants, the double phosphoric acids, act alike with the two bodies. The precipitate formed by picric acid with theobromine appeared to consist of a double chloride; on further investigation it did not prove suitable for effective separation on account of its solubility relations. The precipitation with theobromine which takes place with either HgCl_2 or silver solutions is easily explained on comparing the constitution of theobromine with that of caffeine. The former possesses a labile H atom which can be replaced by one of a metal. In the conversion of theobromine into caffeine this is made use of as a stepping-stone, the metal atom being eventually replaced by a CH_3 group.



As these metallic compounds were apparently likely to be suitable for effecting a quantitative separation, the properties of the lead and copper derivatives were also examined, but these were found unsuitable for the purpose, since they were both soluble. The mercury derivative only separated slowly, and in the condition of a fine powder; there remained, therefore, only the silver compound, and this was found completely suitable for the required purpose.

AgNO_3 , when added to an ammoniacal solution of theobromine, gave at first a flocculent whitish precipitate, which redissolved on the addition of ammonia in excess. On prolonged boiling, as soon as the ammonia had been driven off, the solution became turbid, and eventually a crystalline body was precipitated, which the author considered to be the Ag substitution compound of theobromine. It was insoluble in

water or dilute alcohol, easily soluble in ammonia or dilute HNO_3 . It was found to contain no HNO_3 , and must therefore be a substitution compound and not an additive one; 0.2174 gramme of the dry compound yielded 0.1083 gramme AgCl = 0.0815 gramme Ag.

Calculated for $\text{C}_7\text{H}_7\text{AgN}_4\text{O}_2$	37.63% Ag.
Found	37.488% Ag.

Schmidt has described a silver compound of theobromine containing $1\frac{1}{2}$ molecules of water of crystallization; that obtained by the author suffered no loss in weight on prolonged heating at 130°C .

Caffeine, when treated under the above conditions, gave no reaction, and this is readily explained by its not containing a replaceable H atom. The method, therefore, seemed suitable for the separation of the two alkaloids. In order to ascertain the course of the process quantitatively, the author dissolved a known weight of theobromine in ammonia solution, heated to boiling, added AgNO_3 , and continued the boiling until the NH_3 was driven off and the precipitate ceased to increase, filtered off, washed the precipitate with boiling water until the washings were free from AgNO_3 , dried and incinerated filter and precipitate, and weighed the residual silver. Several unsatisfactory experiments showed that in order to ensure success a large excess of AgNO_3 must be used, and that the boiling must be continued until the whole is reduced to a few c.c. Under these conditions good results were obtained, as is shown by the following examples:

1. 0.1195 gramme theobromine yielded 0.0723 gramme Ag, equivalent to 0.1200 gramme theobromine.
2. 0.1570 gramme theobromine yielded 0.0942 gramme Ag, equivalent to 0.1564 gramme theobromine.
3. 0.1432 gramme theobromine yielded 0.0870 gramme Ag, equivalent to 0.1444 gramme theobromine.
4. 0.2394 gramme theobromine yielded 0.1435 gramme Ag, equivalent to 0.2382 gramme theobromine.
5. 0.1165 gramme theobromine yielded 0.0698 gramme Ag, equivalent to 0.1159 gramme theobromine.
6. 0.1240 gramme theobromine yielded 0.0742 gramme Ag, equivalent to 0.1232 gramme theobromine.

The quantity of theobromine equivalent to that of the silver found can be obtained by multiplying by the factor 1.66 ($108 \text{ Ag} = 180 \text{ theobromine}$).

The result of experiments conducted with mixtures of the two alkaloids are equally good:

1. 0.1075 gramme theobromine yielded 0.0645 gramme Ag, equivalent to 0.1071 gramme theobromine.
2. 0.0862 gramme theobromine yielded 0.0517 gramme Ag, equivalent to 0.0858 gramme theobromine.
3. 0.0911 gramme theobromine yielded 0.0543 gramme Ag, equivalent to 0.0901 gramme theobromine.
4. 0.0792 gramme theobromine yielded 0.0479 gramme Ag, equivalent to 0.0795 gramme theobromine.

Further experiments, in some of which the silver compound was collected and weighed in a tared filter, in others where it was dissolved in dilute HNO_3 and the Ag determined as AgCl , yielded equally satisfactory results. In order to shorten these somewhat tedious processes Volhard's volumetrical method was employed in the following manner. A measured quantity of AgNO_3 solution of known strength was added to the solution of the alkaloids, and, after the precipitation and washing, the residual silver in the filtrate and washings was titrated by ammonium sulphocyanate solution, which had been previously titrated against the AgNO_3 solution.* The actual titration was performed in the cold solution, with the addition of 5 c.c. of a cold saturated solution of ferric ammonium sulphate as indicator, and HNO_3 , which had been boiled until it was colourless. This method gives most satisfactory results; it can be quickly performed, and possesses the advantage that the alkaloids can, when necessary, be recovered and subjected to further examination. For this purpose the silver theobromine is dissolved in dilute nitric acid, the solution neutralized and evaporated to dryness; the caffeine solution, in which the titration took place, is also neutralized and evaporated to dryness. The alkaloids can then be easily extracted from the respective residues by chloroform.

The author considers that he has completely solved the problem of the estimation of the cocoa-alkaloids, and that this result cannot fail to influence considerably the future technical valuation of cocoa and its preparations.

W. J. S.

Estimation of Citrate-soluble Phosphoric Acid in Basic Slag and untreated Mineral Phosphates. P. Wagner. (*Chem. Zeit.*, 1894, xviii., 1153-1154.)—The author insists upon the necessity for a guarantee as to the degree of solubility of the phosphoric acid in basic slag, since, either on account of the low basicity of the slag-phosphate, or by reason of the presence of mineral phosphate as an adulterant, samples vary very much in respect of this solubility. Taking the manurial value of the basic slag found to give the best results in field experiments at 100, other samples of the same content of phosphoric acid, and of the same degree of fineness, gave yields represented by 80, 60, 50, and even 30.

The best method of determining the solubility of the phosphate is by the use of a solution of acid ammonium citrate; and if the results are to be unimpeachable, the treatment with this solution should be preceded by a neutralization of the basicity of the slag by citric acid. For this purpose the basicity is first determined as follows: 5 grammes of the slag are introduced into a 500 c.c. flask, which is then filled up to the mark with a 1 per cent. solution of citric acid and shaken for half an hour in a jigger; 50 c.c. of the filtered liquid are then titrated with $\frac{N}{4}$ soda solution, phenolphthalein being the indicator. For the actual analysis 5 grammes of the sample are placed in a 500 c.c. flask with 200 c.c. of water, and there are successively

* The silver solution employed contained 5 grammes AgNO_3 in 100 c.c.; consequently 1 c.c. = 0.03176 gramme Ag; 14.7 c.c. ammonium sulphocyanate solution ($\frac{1}{10}$ normal) = 5 c.c. of the above silver solution = 0.1588 gramme Ag, and 1 c.c. = 0.0108 gramme Ag.

added (1) the quantity of 5 per cent. citric acid solution previously found to be necessary to neutralize the slag, (2) 200 c.c. of acid ammonium citrate solution (see below), and (3) water to the mark. The mixture is shaken in a jigger for half an hour and filtered; to 50 c.c. of the filtrate 100 c.c. of molybdic solution (see below) are added; the liquid is heated to 80° C., and after cooling the precipitate is filtered and treated as usual.

The ammonium citrate solution is made by dissolving 160 grammes of citric acid and ammonia, equivalent to 28 grammes of nitrogen, in a litre of water. The molybdic solution is prepared by dissolving 125 grammes of molybdic acid in a slight excess of 2½ per cent. ammonia solution, adding 400 grammes of ammonium nitrate, diluting to 1 litre, and pouring the solution into 1 litre of nitric acid (1.19 sp. gr.). After having been allowed to remain at 35° C. for twenty-four hours the solution is filtered.

If it be desired to save the half-hour requisite for determining the basicity of the slag, the analysis may be conducted as described above, with omission of the citric acid. In this case an acid ammonium citrate solution containing 150 grammes of citric acid and ammonia equivalent to 23 grammes of nitrogen must be used. The results are less accurate than when the slag is neutralized. A. G. B.

Milk. J. Lehmann and W. Hempel. (*Arch. Physiol.*, 1894, lvi., 558, through *Chem. Zeit.*)—One of the best distinctions between human milk and cow's milk resides in the nature of the casein. Casein from cow's milk contains 6.6 per cent. of calcium phosphate, whilst that from human milk contains 3.2 per cent. The mean ash content of casein from cow's milk is 7.2 per cent. The ash contains:

Per cent.							Per cent., Calculated on Anhydrous Casein.
CaO	...	49.8	= 3.20
K ₂ O	...	0.9	= 0.06
MgO	...	2.1	= 0.10
Na ₂ O	...	0.4	= 0.03
P ₂ O ₅	...	45.0	= 2.92
SO ₃	...	1.2	= 0.08

It appears that the whole of the phosphorus in the casein molecule is present in the form of a phosphoric acid, and two series of researches gave respectively 1.18 per cent. and 1.50 per cent. of P₂O₅ in the casein molecule. It further appears that casein is a double compound of calcium casein and calcium phosphate in the proportion represented by the formula Ca₃(PO₄)₂, Ca Casein. The proportion of calcium phosphate was not so high in another series of determinations.

The mean ultimate composition of casein is, per cent.:

Ash.	C.	H.	N.	P.	S.
6.47	50.86	6.72	14.63	0.81	0.72

The ash is nearly all calcium phosphate. The ash-free casein contains, per cent.:

C.	H.	N.	P.	S.
54	7.04	15.06	0.847	0.771

The authors do not doubt but that cow's-milk casein and human-milk casein are two individual substances; for not only do they differ in content of calcium phosphate, as shown above, but also in sulphur content, the casein of human milk containing 1.09 per cent. of this element.

The mean results of the many analyses of cow's milk and human milk made for the purposes of this research are as follows:

					Cow's Milk. Per cent.	Human Milk. Per cent.
Casein	3.0	1.2
Albumin	0.3	0.5
Fat	3.5	3.8
Milk-sugar	4.5	6.0
Ash	0.7	0.2
Water...	88.0	88.5

Care was taken that the women were healthy, and that their glands were fully milked.

The coagulation of cow's milk and woman's milk by acids is characteristically different. Whilst the precipitated casein in the cow's milk collects in flocks and sinks to the bottom of the vessel, that in human milk forms a coagulum which does not settle. This difference is traced to the fact that the proportion of fat to casein in the precipitate from cow's milk is so much lower (1.16 : 1) than that in the precipitate from human milk (3 : 1). By the addition of fat to cow's milk the behaviour of human milk when acid is added can be imitated. Dogiel's statement that acids precipitate the casein in coarse flocks from salted human milk is not substantiated.

In order to make an artificial human milk which shall resemble the natural secretion as closely as possible, cow's milk should be diluted with water until its casein content is identical with that of human milk, and cream, milk-sugar, and white of egg should be added until the mixture contains the proportions of fat, sugar, and albumin in human milk. The white of egg should be added in somewhat larger proportion if the suckling is to be fed from the first, since human colostrum, like that of the cow, is particularly rich in albumin.

A. G. B.

Determination of Silicon and Aluminium in Iron. L. L. de Koninck. (*Stahl und Eisen*, 1894, xiv., 138, through *Chem. Zeit.*)—The metal is treated with nitric acid or aqua regia, and precipitated by means of ammonia or by ammonium acetate should manganese be present. The precipitate is dried, ignited, placed in a platinum boat and heated in a stream of hydrochloric acid, whereby ferric chloride is volatilized and a residue of alumina and silica, together with any titanate or chromic oxide that may be present, is left. The separation of the substances thus freed from the bulk of the iron is then effected by the usual methods.

B. B.

EXTRACTS FROM THE EVIDENCE GIVEN BEFORE THE SELECT
COMMITTEE ON FOOD PRODUCTS ADULTERATION, ON
JULY 11, 18 AND 25.

MR. RICHARD BANNISTER.

1. EVIDENCE REFERRING TO THE WORK DONE AT SOMERSET HOUSE, AND THE RELATION BETWEEN PUBLIC ANALYSTS AND THE REFEREES.

542. You have a lengthened experience in the Government laboratory?—I have been there for thirty-one years.

543. And in that Government laboratory you receive cases for analysis, or specimens for analysis, from different localities?—Yes; both for our ordinary official work and also for our work under the Sale of Food and Drugs Act.

544. Under the twenty-second section of the Sale of Food and Drugs Act?—Yes. If you will allow me, I will tell you what we received last year. From the Customs Department we received 2,137 samples; from the Admiralty, 73; from the Board of Trade, 557; from the India Office, 707; from the Post Office, 43; from the War Department, 18; from the Trinity House, 86; from the Stationery Office, 4; from the Office of Works, London and Dublin, 33; from the Colonial Office, 2; and references from magistrates, 71.

545. That is under the Sale of Food and Drugs Act?—Yes; and parochial samples 15, making together 3,889.

546. Of which how many were under the Sale of Food and Drugs Act?—Seventy-one. Then, from the Inland Revenue Department we received 12,083 samples of tobacco; beer for the estimation of drawback, 9,102; beer duty samples, 8,621; beers from public-houses, 2,820; tinctures, 5,575; naphtha, 773; miscellaneous, 5,392.

547. What is the total number of samples?—48,255.

548. I believe you have a table of specimens referred to you under the Sale of Food and Drugs Act from 1875 to 1894?—Yes.

549. Can you give us a brief analysis of that table?—Yes, I can. Samples of arrowroot, 4; beer, 5; bread, 7; brandy, 4; butter, 62; carbolic acid, 1; coffee, 26; cream, 1; flour, 3; gin, 6; ginger, 5; gingerbeer, 1; ketchup, 1; lard, 23; lemonade, 3; laudanum, 1; linseed-meal, 1; magnesia, 1; milk, 411; mustard, 8; sweet nitre, 4; oatmeal, 13; ointment, 1; peas, 2; pepper, 29; quinine, 4; rum, 3; soda-water, 1; tea, 3; vinegar, 14; *water*, 2; whisky, 28. Total, 678.

550. In those 678 specimens submitted to you, in how many did you agree with the previous analysis?—In the case of 474.

551. And you disagreed in how many?—In the case of 188; and 16 samples were sent up for a special purpose, which were not entirely sent up under the Sale of Food and Drugs Act strictly.

554. Milk and butter were really the two principal articles that were submitted to you?—Exactly.

1609. I notice that you mentioned two samples of water referred to you under the Sale of Food and Drugs Act: what water would that be?—That would be water referred to us under the Public Health Act by the magistrates.

1610. I notice that the Sale of Food and Drugs Act expressly excludes water?—Yes; we do not make a separate return of milk and water, but those samples were referred to us by the magistrates under the Public Health Act.

1611. With regard to the cases that you mentioned of your analyses differing from those of the public analysts, I think you said that in 474 cases you agreed, and that you disagreed in 188 cases: is that so?—Yes.

1612. Could you tell us whether this disagreement took place in the earlier stages of the working of the Sale of Food and Drugs Act?—I have not got the returns for every year, but I think I have got the return for the last three or four years here; and I looked at that, and thought that there was very little difference.

1613. Why I ask you is, that I notice in one of your reports it says that a year or two ago you had 47 samples submitted to you, and that you disagreed only in two cases, so that it occurred to me that the disagreement was lessened.—There is no doubt that it was up to a certain time. I think that last year there was a little difficulty about margarine; but, if I remember rightly, there was a steady decrease.

1614. That is to say, a steady decrease in the number of disagreements between Somerset House and the public analysts?—Yes.

1615. I suppose it necessarily followed that some of the samples submitted to public analysts would be wrongly reported upon by them?—No doubt.

1616. But in the general working I presume you are of opinion that the public analysts have done their work efficiently?—I think that during the last six or eight years of the working of the Sale of Food and Drugs Act the work has been done remarkably well.

1617. And those differences that have cropped up between you have been differences rather more as to the interpretation of results than actual mistakes made?—Yes.

1618. That is to say, that from the same analytical results two analysts, both working accurately and honestly, may, and very frequently do, arrive at different conclusions?—They do.

1619. But, taking your figures from the last twenty years, the average differences between the public analysts and Somerset House have not amounted, as far as my calculation takes me, to more than, on an average, ten cases a year.—I dare say that is right.

1621. In the results which you have given us of samples that have been referred to you there were included samples from Scotland and Ireland, as well as from England, I believe?—There were.

1622. But the Reports of the Local Government Board, as I understand them, only refer to samples taken from England under the Sale of Food and Drugs Act?—I cannot answer that question as regards their return; one return goes in for the whole of them. So far as the references are concerned, it goes in for England, Scotland and Ireland.

1623. But I have looked at it rather carefully, and I make out that the Local Government Board's report refers only to samples from England. The average of the samples taken and submitted to the public analyst since the working of the Act would be about 25,000 per year, roughly speaking, I believe?—I should say quite that.

1624. And you disagreed, on an average, with regard to one sample in 2,500?—That is in the reference.

1625. I would like to clear up one reply of yours here, in answer to question 556, which may be subject to misconception, although I think I understand it clearly. You stated that you disagreed in about one quarter of all the cases: you do not mean in one quarter of all the samples taken, but only in those submitted to Somerset House as a court of reference?—My answer had nothing to do with the others, so that I hope that my remarks cannot be misunderstood.

1626. There were sixteen samples, you told us, sent up for a special purpose, not sent up under the Sale of Food and Drugs Act strictly; what were those sixteen samples?—I cannot give you the particulars of them, but they were samples arising out of previous examination; they were not really confined to samples that had been referred by magistrates from the defendant, but were referred for some special information that the magistrate wanted.

1627. You have had at Somerset House, outside the references that have come to you under the Sale of Food and Drugs Act, a large experience with milk and butter samples?—Yes.

1628. Would that be an experience as large and varied as that of the public analysts?—The experience would not be so large as that of the public analysts, because they are generally examining milk from morning to night.

1629. But Somerset House is not continually engaged in making examinations into a large number of samples of butter and milk?—We are continuously engaged in getting accurate results of examinations.

1630. But in a general way should I be overstating the case were I to suggest that the experience of the public analysts, so far as butter and milk are concerned, is larger than that of Somerset House?—So far as the actual examination of samples is concerned, yes.

1631. And of course the public analysts have their general practice, in addition to the work that comes to them in their official capacity?—Certainly.

1632. They are analytical chemists with a private practice, many of them, I presume?—Yes.

1633. There is going on, I understand, between public analysts an interchange of ideas, and experiences, and opinions in connection with analytical science?—No doubt.

1634. And, therefore, would it be fair to assume that they act upon their collective experience, or, to put it in another way, that the best information that the best of them may procure is placed at the disposal of other analysts throughout the country?—That is so to a large extent.

1638. I suppose, from time to time, certainly since the passing of the Act, there has been a great improvement in the science of food analysis?—No doubt.

1639. And the public analysts have contributed very largely to this improvement?—Certainly.

1640. Somerset House, I presume, gets the benefit of that improved knowledge acquired by the public analysts, and avails itself of the information?—Yes; but, of course, in our investigation of food analysis we always try as much as possible to see

for ourselves, by direct experiments, the value of the results reported, and of the different methods of analysis.

1641. That means, naturally, that you do not necessarily accept in all cases the deductions, or improved results that the analysts announce as having been arrived at?—Exactly.

1642. *But in the case of Somerset House arriving at any improved result, or differing in their own minds from some new result, does Somerset House feel it incumbent upon itself to put itself into communication with the public analysts, either to say: Here we have discovered a new method that ought to be known throughout the country, or, We have subjected this new method of yours to close investigation in our laboratory, and we do not agree with the results which you inform us you have arrived at?—We do not make it a general practice, but with some public analysts, they are in direct communication with us, and, of course, we interchange ideas and opinions about different methods of analysis.*

1643. May I say, then, that there is a close intercommunication continuously being carried on between the public analysts of the country and Somerset House?—With some of the public analysts.

1644. But not with the Society of Public Analysts in its corporate capacity?—No. But I can give you a simple illustration of that: When poivrete was being largely used in the adulteration of pepper, one public analyst had a difficulty in separating it so as to show it to the magistrate, and I had the pleasure of showing him exactly how it could be done, and from that time forward the adulteration of pepper with poivrete has ceased completely.

1645. Because the public analyst received this information from you?—He not only could say from its analytical nature that poivrete was there, but he could show the magistrate its presence.

1646. Showing the enormous advantage to the country from that revelation by Somerset House?—Certainly.

1647. And, I suppose, that Somerset House deems it necessary that there should be this interchange of opinion between it and the public analysts?—*It is very desirable.*

1648. And may I understand that it is being carried on on a systematic basis, or is it merely casual?—It is on a systematic basis with some members of the Society of Public Analysts.

1649. With those of the most repute, as far as regards reputation?—Yes, men of repute.

1650. And those men are in close communication with Somerset House?—Yes.

1651. Interchanging ideas and receiving suggestions from you, and conveying suggestions to you?—Yes.

1652. Have you ever conveyed to the public analysts the details of the methods used by you, or of any standards or limits that you use?—As soon as we made an investigation into the composition of food products, we published the result of the investigation in two little manuals; we made them general property.

1653. Such as Dr. Bell's little work?—Yes.

1657. How many years is it since you issued a manual, do you think, from which the public analysts might gain the advantage of your improved experience?—

The years I cannot tell you, but you will see it from the two manuals from the title-page.

* 1658. But there is no systematic publication to which the public analyst can look for inspiration from Somerset House?—There is not.

826. How do you make your standards known?—If any gentleman writes about it we send him word.

827. *You do not communicate it authoritatively to the public analysts?—We do not consider that we have authority to do anything of the kind, because they are an independent body altogether, and they might think it was dictation. We do not withhold it, but we do not communicate it.*

828. *But it becomes known through test cases, does it not?—Yes.*

2. EVIDENCE RELATING TO MILK.

555. Will you tell us now with reference to the milk cases—will you give us the analysis of the milk cases that came before you? And 552. I notice that you have had a far larger number of milk specimens than of anything else.—Yes; we had 411. In 311 cases we confirmed the decision of the public analysts, and in 96 we disagreed, which is a percentage of disagreement of 23·5.

556. So that in not quite a quarter of the cases you disagreed with the public analysts of the country?—Yes.

557. Could you refer this difference to any particular cause or condition?—I think that the difference is rather more in the interpretation of the results than in the actual mistakes made.

558. That is to say, that the analyses made by the public analysts were probably correct, but that their interpretation of the analysis differed from yours?—We differed in the conclusions.

559. In what way would this difference of interpretation arise?—It would arise when we came to the border-line between a genuine milk and an adulterated milk, or, as put in another way, between a good milk and a poor milk.

560. That is to say, that a poor natural milk was occasionally mistaken for adulterated milk?—I think I have not made myself quite clear. It is practically impossible from the analysis of a sample to tell whether it is poor milk or an adulterated milk—that is to say, whether it has been a good milk adulterated with water, or a poor milk.

561. *It really comes to this: that a naturally poor milk has been reported as an adulterated milk?—As a rule that is the case.*

562. And that gives rise to the difference between you and the public analysts to a considerable extent?—Mainly.

563. It accounts largely for the 96 cases in which you disagreed?—Yes.

564. Can you tell us whether you have any methods on which you rely for arriving at that conclusion; any special tests?—The process that we use at Somerset House, after trying every process that we have known, or that has come before us, is what is called the maceration process. (The witness then described the process as laid down in Dr. Bell's book.)

568. That is the process that you rely upon for estimating the natural condition of milk?—Exactly. *Then, in the examination of a sample, there are certain physical conditions which we have to take into consideration as well, and all these conditions, along with the result of the analysis, are taken together for the purpose of forming a conclusion as to whether water has been added or not.*

569. Are you able to arrive at a conclusion with moderate certainty as to whether the milk is a poor milk or watered milk?—We are not.

570. That is to say, that you cannot distinguish between a rich milk which has been diluted and a milk coming from a poorly-fed or old cow?—*We cannot.*

571. There are different methods of analysis which give somewhat different results, are there not?—Yes, there are.

572. Do those results vary widely?—Not if the examination is conducted properly. For instance, if the coil process be used it will, perhaps, give an increase of what is called butter-fat of perhaps 0.2.

581. So that when you have any analysis before you, or when the magistrate has any analysis before him, it is necessary that he should consider the method by which the results have been arrived at?—*He ought to do so.*

1707. Has he any opportunity of ascertaining this important difference of process?—There is no doubt about it that if we are coming to a small admixture of water *it is necessary* that the method of analysis should be known.

1708. But you do not, as a matter of fact, state on your certificates the process which you follow?—*No.*

583 and 584. Can you give us a description of some experiments that you made with 273 cows?—We obtained these samples of milk from different parts of the country and at different times of the year. You will see in the return that everything is given about the feeding of the cows, so that nothing can be said to the effect that the cows were ill-fed; and we sent one of our own reliable assistants down, so that he saw the cows milked out completely, and the milk was always under his supervision until he got the samples, and had brought them to our own laboratory for examination. In that return we give 273 cases of individual cows, and also the results of the examination of 55 churns. (586) We examined all these samples by the maceration process, and we found that of non-fatty solids the lowest was 7.52, the highest 10.04, and the average 8.90; and so far as the fats are concerned the lowest was 2.43 in two cases, the highest 5.97, and the average 3.99. Then, from the 55 churns the lowest was 8.40, the highest 9.70, and the average 8.96; and of the fat solids the lowest was 2.89, the highest 5.61, and the average 4.00.

591. What is your standard?—When I am talking of the standard I ought to have a word presently about the difference between the standard and the limit. The standard that we are speaking of, and can call the standard *for the present*, is 8.5 solids-not-fat.

593. You referred to a standard and a limit just now. Will you just define what you mean?—The difficulty about a standard and a limit arises in this way: Under the Adulteration of Foods Act, 1875, *it is laid down distinctly that if you have milk, or any other substance, from which nothing has been taken away, and to which nothing has been added, however poor, it is genuine.* In the case of milk, a large

quantity has to be sold in towns quite away from the place of production, and we do not know its origin; therefore, when we get a sample of milk of low quality, the analysts and ourselves (and I think it very proper that they should do it) fix a limit, so that if there is any milk that falls below that limit, *it is for the person to prove or give conclusive evidence to the satisfaction of the court that water has not been added.*

1701. In your opinion, ought everything that passes from the cow be defined as pure milk?—It is not a matter of opinion with me; it is the interpretation of the law.

1702. And you interpret the Act in that way?—Exactly.

1703. That it leaves you no option but to declare everything as pure to which nothing has been added, and from which nothing has been abstracted?—*That is the wording of the Act*, and we are bound by that.

1704. Can you point out in the Act where that wording is referred to; I have looked through it carefully, and cannot quite follow it?—The interpretation is from section 6: “No person shall sell to the prejudice of the purchaser any article of food, or any drug which is not of the nature, substance, and quality of the article demanded by such purchaser, under a penalty not exceeding £20,” and so on.

1723. On this question of your construction of the Act, bearing on the point of poor milk being accepted as being pure milk, if a vendor of milk supplied what is termed fore-milk, which is almost free from cream, would you not consider that the purchaser would be prejudiced in such a case?—I think he would be.

1724. Would not the Act apply then?—I think that is for the magistrate to determine whether the Act applies or not. Personally, if I were buying that milk, I should think I was prejudiced.

1725. Can you tell us whether that opinion has been held in courts of law?—I cannot tell you.

1726. I think there have been cases where a vendor has been selling milk from a churn, where one purchaser has come to buy who has received practically the cream at the top through carelessness on the part of the vendor, and another purchaser has come and has received what is practically skim-milk; and I believe the courts have held that the purchaser has been prejudiced in the latter case?—Yes; I should say that the court will settle it in that way.

1727. But still, at Somerset House you maintain the opinion that, notwithstanding that the milk is very poor, provided that it comes from the cow, it is a legal tender, if I may so use the word—it is saleable as a pure article?—I think it is scarcely right to say that we hold that opinion at Somerset House. I would rather put it in this way, that the interpretation of the law seems to be in that direction; but so far as we at Somerset House are concerned, the only matter that we have to settle is to examine the sample of milk that comes before us.

594. How is the limit (for milk) fixed?—It has been fixed on the part of the public analysts in this way: that when the Act of 1872 came into force they took the figures of Mr. Wanklyn, and *Mr. Wanklyn's figures were 9·0 and 2·5*; that is to say, 9·0 of solids-not-fat, and 2·5 of fat.

595. Then they adopted that as their limit?—They adopted that as their standard. Then in 1882 it had been found between those two years that by the method of analysis the solids-not-fat did not give a constant weight; and so in a

paper read before the Society of Public Analysts on March 15, 1882, these facts were brought before the Society: "It can hardly be expected that anything very novel can be brought forward on a subject which has been so well ventilated, and which before all others has engaged the attention of public analysts, as milk analysis" (the witness quoted from a paper on page 60 of THE ANALYST, vol. vii., to the end of the first paragraph, and then continued). Then the paper goes on, and it is shown distinctly that as soon as ever you dry to a constant weight of 9.0, in which the sugar of milk is present in a crystalline form, it comes down to 8.5 because it is anhydrous, and, therefore, when the drying is to a constant weight of 8.5, it is equal to the old 9.0.

596. You have given the decrease of the solids-not-fat; would you give the increase of fat?—*There is no increase of fat mentioned in the paper, and I shall be able to explain, if necessary, how that has come about.*

597. You had better explain that, perhaps?—*In going further with this paper, there is no difference at all given in the increase of fat, the fat remains at 2.5.*

598. And the total solids-not-fat diminished?—*The solids-not-fat diminish from 9.0 to 8.5, and the explanation is given in a subsequent paper of Mr. Hehner's; he finds that this is due to the fact that the milk-sugar that is present in the milk, when dried only for that time, does not give up its water of crystallization, but that as soon as it is dried to a constant weight then water is given up which is equivalent to about five-tenths of a per cent.*

1714. Does not Mr. Hehner state in that very paper that whilst diminishing the solids-not-fat they transfer that half point diminished to solids that are fat?—*You have the book before you; will you kindly tell me where that appears?*

1715. It is at page 64; "*and in every instance do the modifications in the methods of analysis which I have examined tend to yield a lower amount of solids-not-fat, and a larger percentage of fat, than does the original (Wanklyn) method.*"—*The point which I raised was this, that there was no increase by the public analysts in the percentage of fat from 2.5 to 3.0 as suggested.*

1716. I understood you to say that there was no increase of fat absolutely suggested?—*No. The method of analysis that has been spoken of by Mr. Hehner is a different method from the Wanklyn method; it is one where you bring in the Soxhlet extractor, and that is well known to have a tendency to increase the amount of weight of that substance that is weighed as fat; but there is very little doubt that there is something there besides fat.*

(NOTE.—Our readers are recommended to turn to the papers in THE ANALYST referred to by Mr. Bannister, page 60, vol. vii., and page 253, vol. viii., and to satisfy themselves how far Mr. Bannister's statements are warranted by the contents of the papers in question.)

(To be continued).

CORRESPONDENCE.

To the Editors of THE ANALYST.

4, New Court, Lincoln's Inn, London, W.C.

August 30, 1894.

SIRS,—The following details of a prosecution for selling adulterated milk may be of interest to your readers.

The sample was analysed by me, and found to contain, as the mean of two very closely concordant results :

Total Solids	11·10
Fat	2·89
Solids-not-Fat	8·11
Ash	0·678

I certified that it contained 5 per cent. added water. When the case was heard, Mr. De Hailes gave evidence to the effect that, though the milk was of poor quality, it was not adulterated.

The sample was referred to the Somerset House authorities. The following is their analysis :

Non-fatty Solids	8·25
Fat	2·62
Water	89·13

They said : " From a consideration of these results, and after making addition for natural loss arising from the change which occurred in the milk through keeping, we are of opinion that the milk, although of poor quality, does not afford conclusive evidence of added water.

(Signed) " R. BANNISTER,
" J. HOLMES."

As is almost invariably my experience, the fat found was considerably below that found by myself, and the solids-not-fat are consequently too high.

When the Somerset House chemists analysed the milk, it was at least twenty-eight days old. Allowing for some natural loss in the solids-not-fat by decomposition, the fat would, according to their results, have been less than 2·62. Even 2·62 is below what Mr. Bannister informed the Select Committee was their standard for fat—viz., 2·75—and yet not a word is said in the certificate that the sample was deficient in fat.

The County Council of Middlesex applied for an adjournment, but without success. In view of Mr. Bannister's statement to the Select Committee, that when a milk falls below the standards, the responsibility of proving it to be genuine should lie with the vendor, it seems to me to be a pity that he did not intimate as much to the magistrates.—I am, yours faithfully,

EDWARD BEVAN.

NOTICE.

In recognition of the services of the late Dr. Arthur H. Hassall, Lord Rosebery has granted a pension of £50 per annum from the Civil List to Mrs. Hassall.

THE ANALYST.

OCTOBER, 1894.

ON EXTRANEOUS MINERAL MATTER CONTAINED IN COMMERCIAL GINGER.

By ALFRED H. ALLEN.

In the course of examination of samples of ginger for the detection of an admixture with the previously extracted article, I have occasionally met with samples containing such a large proportion of mineral matter as to suggest strongly an intentional adulteration with warehouse sweepings. To those familiar with the practice that requires drug-grinders to return the full weight of an article sent to them to grind, the presence of such an impurity will excite but little astonishment, while, of course, there will be no difficulty in finding among the sympathisers with adulteration defenders of such a "trade practice."

I have recently met with two samples of ground ginger to which, in my opinion, exception may fairly be taken on the above ground; and in one instance I felt it my duty to certify to the presence of "not less than 5 per cent. of sand and extraneous mineral matter." The sample in question showed on duplicate analysis the unprecedented proportion of 10.6 per cent. of total ash, of which 2.3 per cent. was actual sand insoluble in acid. On agitating the sample with chloroform nearly 8 per cent. of mineral matter mixed with some organic matter was deposited, and, of course, the mineral matter thus separated cannot be natural to the ash of the ginger. The proportion of ash soluble in water, and the constituents of the ash also pointed to the presence of a notable quantity of extraneous mineral matter besides actual sand.

But the excessive proportion of total ash yielded by the sample is itself sufficient to establish the substantial accuracy of the conclusion at which I arrived, as is evident from a consideration of the following summary of the proportion of ash yielded by 104 specimens of ginger. Of these, 34 are analyses of ginger recorded by other chemists, and are all the published results with which I am acquainted. The figures yielded by Japanese ginger are given in order to complete the data; but this variety is rather a curiosity than an article of ordinary commerce, and ought not to be taken into consideration when interpreting the results.

PROPORTION OF ASH YIELDED BY 104 SAMPLES OF GENUINE AND COMMERCIAL GINGER.

Description of Ginger.	Maximum.	Minimum.	Average.	Number of Samples.	Observer.	Reference.
Ginger Roots ...	3.90	3.29	3.66	7	Allen and Moor.	<i>Analyst</i> , xix., 126.
Ginger Roots ...	7.02	3.39	4.72	6	C. Richardson.	<i>U. S. Bulletin</i> , No. 13.
Ginger Roots ...	4.10	3.10	3.76	6	Dyer and Gilbard.	<i>Analyst</i> , xviii., 197.
Ginger Roots ...	5.00	3.10	4.04	5	T. H. Pearmain.	<i>Analyst</i> , xix., 125.
Japan Gingers ...	6.58	3.34	5.02	3	B. Dyer.	<i>Analyst</i> , xix., 127.
Ground Gingers ...	8.00	3.40	5.00	7	W. C. Young.	<i>Analyst</i> , ix., 214.
Ground Gingers ...	7.94	3.45	5.54	7	C. Richardson.	<i>U. S. Bulletin</i> , No. 13.
Commercial Ground Gingers ...	10.65*	2.48	4.42	63	A. H. Allen.	Samples under Food Act.
General Average ...	10.65*	2.48	4.46	104	Various.	

From the foregoing table it appears that genuine unground ginger-root yields barely 4 per cent. of ash on the average, and only in a single specimen (recorded by C. Richardson) did the ash rise to 7 per cent. Of the 63 samples of commercial ground ginger purchased by inspectors and submitted to me under the Sale of Food and Drugs Act, only two samples contained more than 8 per cent. of ash, one of these being the sample already referred to, while the other contained upwards of 3 per cent. of sand. But, even including these, the average ash of the 63 samples was only 4.42 per cent., while the general average of all the 104 samples examined was but 4.46 per cent. Of course, the contention that genuine ginger may contain 10 per cent. of mineral matter, natural and extraneous, would, if successful, enable the dishonest trader to make at least 5 per cent. of illicit profit on nearly all ginger which passed through his hands.†

On receiving my certificate on the sample of ginger yielding upwards of 10 per cent. of ash, the West Riding authorities issued a summons, and in due course the case was heard. In support of my certificate I put forward the above statistics.

For the defence, Mr. F. M. Rimmington, Public Analyst for Bradford, stated that he had analysed the defendant's portion of the sample, and recognised it as Barbadoes ginger, which was a low quality. The proportion of total ash, he stated, was not excessive, and the "silex" present was of "underground origin," and natural to the ginger. Mr. Rimmington admitted that he had not looked for any extraneous mineral matter other than sand. At this point the magistrates announced that they had decided to refer the case to Somerset House. The referees in due

* This was the adulterated sample already referred to. The next highest sample on record contained 8.18 per cent. of total ash, which included upwards of 3 per cent. of sand.

† Since writing the above I have met with an analysis by Mr. E. W. T. Jones (*ANALYST*, xi., 75) of a single specimen of ginger which was found to yield 4.80 per cent. of ash on ignition.

course gave a certificate stating that the sample contained 8.82 of mineral matter, of which 2.11 was sand. They went on to certify that "the total amount of sand and other mineral matter present in the sample is high, and would be regarded as excessive if found in a high-class ginger which had been washed before being ground; but the results obtained are not greater than are sometimes found in low-priced ginger which has been ground as imported. We are of opinion that the sample in question affords no evidence of the presence of sand or extraneous mineral matter other than that present in unwashed ginger."* This certificate was signed by Messrs. R. Bannister and H. J. Helm. The Bench held that if people wanted cheap ginger they must expect to have with it a certain amount of dirt, and dismissed the summons, with costs.

I do not think that the difference in the proportion of ash reported by the referees and that found by me is due to any error of analysis, since other chemists have obtained figures some of which agree with those of Somerset House, and others are more nearly in accordance with those obtained by me. In my opinion, the variation is more probably due to the extraneous mineral matter having gravitated to a particular part of the sample.

Since the occurrence of the foregoing interesting case, Mr. W. F. K. Stock has communicated to me the results yielded by 58 samples of commercial ground ginger analysed in his laboratory. He found

28 samples to yield between 3 and 4 per cent. of ash

19	"	"	4	"	5	"	"
4	"	"	5	"	6	"	"
5	"	"	6	"	7	"	"
1	"	"	7	"	8	"	"
0	"	"	8	"	9	"	"
0	"	"	9	"	10	"	"
1	"	"	10	"	11	"	"

Of these 58 samples, 51, or 88 per cent., yielded less than 6 per cent. of ash. The maximum was 10.86 and the minimum 2.90 per cent. These samples were bought at intervals over a wide district, and hence, no doubt, fairly represent the ground ginger supplied to the public. The sample yielding 10.86 per cent. of ash was the only one of the number showing more than 8 per cent., and was certified by Mr. Stock to be adulterated. Mr. C. G. Moor informs me that he found eight specimens of commercial ground ginger analysed by him to yield ash varying from 2.98 to 7.00 per cent., with an average of 4.55 per cent.

Since the foregoing statistics were compiled I have received for analysis, under the Sale of Food and Drugs Act, a sample of ground ginger giving the high proportion of 11.9 per cent. of total ash. The character of the mineral matter in this case is, however, very different from that previously referred to. Instead of containing sand, the sample in question showed on analysis the presence of upwards of 8 per cent. of

* It has been pointed out by a brother analyst that the presence of sulphates and calcium salts in abnormal proportion indicated that the sample in question had been bleached, and hence that it was not unwashed ginger.

calcium sulphate. On treatment with a considerable quantity of cold water, the ginger yielded a solution which gave copious precipitates with barium chloride and ammonium oxalate, and on weighing these precipitates, produced under proper conditions, the sulphate and calcium thus estimated left no room for doubt as to the combination in which they existed. The interesting question next arose as to the form in which the calcium sulphate had been introduced. If it were ascribable to a badly managed bleaching process by calcium sulphite, the presence of well-formed crystals of gypsum was to be expected. If added as plaster of Paris, these crystals would be formed (as was proved by experiment) on treating the sample with water. But on agitating the sample with chloroform a deposit was obtained which, under the microscope, was seen to consist largely of crystalline fragments of gypsum, having exactly the appearance and optical characters of a sample of fibrous gypsum with which the deposit from the sample was compared. Hence there is no doubt that the sample contained an admixture of the ground mineral, and it is impossible to attribute its presence to accident or natural causes. But 8 per cent. of anhydrous calcium sulphate corresponds to 10·1 per cent. of gypsum ($\text{CaSO}_4 + 2\text{H}_2\text{O}$), so that the adulteration was practised to the extent of fully 10 per cent.

TWO DISPUTED CASES OF ADULTERATION.

By E. G. CLAYTON.

PARTICULARS of the following cases were privately sent to Mr. Hehner a short time ago; but it has occurred to me since that at the present juncture they may be of sufficient interest for publication in THE ANALYST.

1. In 1892 I received a sample of milk, No. —, and certified that it contained 10 parts of added water. The analytical data are here stated:

<i>Found.</i>				<i>Calculated.</i>			
Specific gravity	1028·9	Fat	3·29
Total solids	11·18	Non-fatty solids	7·89
Fat	3·31				
Non-fatty solids	7·87				
Mineral matter	0·675				
Cream	5 per cent.				

The case was referred to the Somerset House analysts, a copy of whose certificate is appended (I omit the dates and sample-number, but the date of receipt at Somerset House was just thirty days later than the date of receipt of the sample by myself):

“The sample of milk referred to in the annexed letter, and marked —, was received here on the —th ult. securely sealed. We hereby certify that we have analysed the milk, and declare the results of our analysis to be as follows:

Non-fatty solids	8·08 per cent.
Fat	3·18 „
Water	88·79 „

"From a consideration of these results, and after making addition for natural loss arising from the change which has occurred in the milk through keeping, we are of opinion that the milk contains not less than 3 per cent. of added water. As witness our hands, this — day of —, 1892.

"(Signed) { "J. BELL, D.Sc., F.R.S.
 "R. BANNISTER, F.I.C., F.C.S.
 "JOHN HOLMES, F.I.C."

At the adjourned hearing in court, in consequence of the fact that the Somerset House certificate (based on an analysis of a sample one month old) appeared to show that the milk was not adulterated to the extent certified to by myself, but only in a slight degree, the case was, on the advice of the magistrate, *withdrawn*.

2. In 1891 I received a sample of coffee, No. —, and certified that it contained 84 parts of chicory. At the hearing the defendant denied having added more than 50 per cent. of chicory (1). The case was adjourned, and referred to Somerset House. The analysts there certified that the sample "contained not less than 33 per cent. of chicory." The magistrate said that the certificate (*i.e.*, the Somerset House certificate) did not say how much more, and was very indefinite; he then inflicted a fine of £3 and 12s. 6d. costs. (These particulars are taken from the newspaper report.)

Although in this case there was no miscarriage of justice, the vagueness of the wording of the referees' certificate was such as to invoke a magisterial criticism. The disputed milk case calls for no comment, its general similarity to numerous others being sufficiently obvious.

The foregoing are the only two disputed cases of which I have a personal knowledge.

The Occurrence of Chlorine in Saltpetre. A Hellich. (*Chem. Zeit.*, 1894, xviii., 485, 486.)—The author examined a sample of saltpetre (which gave indications when tested directly of being almost free from chloride) for bromate by deflagrating a mixture of the salt with sugar, extracting the residue, acidulating and precipitating with silver nitrate. In spite of the previous proof of the absence of chloride in the original saltpetre, the product of deflagration yielded a precipitate of silver chloride. That this chloride was not derived from any impurity in the sugar was proved by deflagrating the saltpetre with starch, cotton-wool, and sulphur instead of sugar, a precaution which did not prevent the occurrence of a notable reaction for chlorine in the deflagrated mass. When the saltpetre was heated alone, at a temperature not much above its fusing-point for a few minutes, no chlorine reaction was developed; but after stronger and more prolonged heating chlorine as chloride could be detected. When the saltpetre was strongly heated with manganese dioxide the indication of chloride was yet more marked. The examination of various samples of saltpetre was then carried out by the following method, adopted by the author as a standard: 10 grms. of the sample were strongly heated with 0.5 gm. of manganese dioxide until the melt assumed a uniform green colour, when it was dissolved in 50 c.c. of distilled

water, decomposed with 20 c.c. of dilute sulphuric acid, allowed to stand for some minutes, filtered and precipitated with silver nitrate. The purity of the reagents was of course ascertained. That the silver precipitate really consisted of silver chloride was determined by numerous tests.

The author has endeavoured to decide in what form the chlorine is present in the saltpetre, as it does not exist as chloride until the saltpetre has been heated, and he has come to the conclusion that it exists as perchlorate, which, in samples giving the reaction strongly, may exist to the extent of 0.25 per cent. The occurrence of this impurity appears to be common even in "chemically pure" specimens. B. B.

The Analysis of Ethereal Oils. J. Klimont. (*Chem. Zeit.*, 1894, xviii., 641, 642 and 672, 673.)—The most rational system for the examination of ethereal oils is that based on the determination of chemical constants depending on the composition of the oil. At present the observation of a few physical properties is commonly made to suffice for the discrimination of these substances. The determination of the acid value, the saponification equivalent, the methyl number and the carbonyl number (*Sitzungsber. d. K. Ak. Wissensch. Wien. Kl. C. II. Abth. II. b.**), constitutes an important step in this direction, and has now been supplemented by the determination of the terebenthene number, which depends on the fact, observed by the author, that direct quantitative addition of bromine is practicable with essential oils. The process has to be conducted differently from the usual method of determining the bromine absorption, which proved uncertain whether direct titration of the essential oil (in chloroform) with bromine solution, or the plan of adding excess and titrating back, was used. The reagents needed for the execution of the author's method are: (1) a solution of bromine in chloroform of about 1 per cent. strength; (2) an approximately pure terebenthene prepared after Ribau by washing commercial French oil of turpentine with soda, distilling and collecting the fraction passing over between 168° and 170°C.: the substance thus prepared can be kept for months in a well-stoppered bottle provided with a glass cap ground on to the neck; (3) chloroform, not necessarily pure, but identical in quality for any given set of experiments. A pure product may, however, preferably be prepared by shaking with strong sulphuric acid, washing and distilling. The bromine solution is standardized by measuring out about 0.5 c.c. of terebenthene (and determining the exact quantity taken by weight) into a 20 c.c. flask provided with a stopper well ground in. The weighed portion of terebenthene is dissolved in chloroform, filled into a small burette, and used to titrate 10 c.c. of the bromine solution to be standardized. The titration is conducted by adding the terebenthene solution little by little and shaking repeatedly until the bromine solution is completely decolorized. The end-point is sufficiently definite. A corresponding titration with the ethereal oil (in chloroform) to be tested is then made in the same way. As the substances capable of combining with bromine under the conditions of the experiment comprise bodies other than terpenes, the terebenthene number does not necessarily represent the percentage of terebenthene in the oil examined, but expresses the total

* No more accessible reference is quoted.—B. B.

bromine absorbed in terms of *terebenthene*. The bromine needs standardizing afresh when more than a day has elapsed since its last standardizing. In order to make the determination of the terebenthene number useful as a means of judging the quality of an essential oil, the author has determined the terebenthene numbers of many samples, the results being given below.

Terebenthene numbers of various oils of turpentine and their adulterants :

Nature of sample.	Terebenthene number.
French oil of turpentine	99.9
American "	101
Austrian "	94.9—97.3
Russian "	70.2—82.5
Hungarian "	76.8
Russian " (deodorised)	80.3—80.9
Rosin oils	52.1—67.6
Petroleum	1.5—1.6

Genuine oils of turpentine (French and American) are distinguished from their adulterants by their high terebenthene number.

Terebenthene numbers of other ethereal oils :

Name of oil	Terebenthene number.	Colour at end of reaction.
Oil of anise	51.5	
Citronella oil	80.0—82.5	
Oil of lemon	117.7—120.2	
„ cinnamon	22.5—23.8	
„ cassia	21.1—40.9	... cherry red
„ rosemary	58.5—59.7	
Angelica oil (seed)	78.4—79.6	
„ „ (root)	97.4	... blue
Oil of peppermint (Japanese)	26.2—27.8	
„ „ (Mitcham)	14.5—18.5	... rose
„ „ (red)	9.8	
„ caraway	99.2—107.7	
Ol. carvi	77.1—79.5	
Curly mint oil (Krauseminzöl)	88.4—89.7	
Oil of fennel	44.9—58.8	
„ orange	119.0—121.1	... lemon yellow
Oil of calamus	67.3	
„ bergamot	94.2	
„ cloves	55.1	
„ cajeput	24.9	
Bajala oil	85.6	
Sandal-wood oil	69.3	
Oil of juniper	105.2	
Oil of cumin	20.7—24.9	... emerald green
Oil of bitter almonds	0.0	
„ rue	10.1	

The author, in commenting upon these figures, points out that the variations in the values obtained for different samples of oil of peppermint are to be ascribed to the state of oxidation (due to keeping) of each sample. The large differences observed in the case of oil of cassia are not readily explicable. At the end of the

paper he insists on the necessity of working a process of this kind, arbitrary as it is, under conditions identical for all samples, and conducting the titration as rapidly as possible, so that any slow secondary absorption of bromine may not be reckoned as that characteristic of the oil under examination. B. B.

Detection of Perchlorates in the Presence of Chlorides, Chlorates, and Nitrates. F. A. Gooch and D. A. Kreidler. (*Zeits. anorg. Chem.*, 1894, vii., 13; through *Chem. Zeit.*)—The authors have experimented on the detection of perchlorates by evaporating various quantities of a solution of potassium perchlorate to dryness and fusing the residue with anhydrous zinc chloride. Chlorine is evolved by this treatment, and can be recognised by causing it to liberate iodine in the ordinary way. As little as 0.05 milligrammes of potassium perchlorate gave a distinct reaction under these conditions. Chlorides have no influence on the process. Chlorates are previously decomposed by evaporation to dryness with strong hydrochloric acid. In order to prevent nitrates interfering, the dry substance is treated with 2 c.c. of a saturated solution of manganous chloride in strong hydrochloric acid. The liquid is evaporated to dryness, and the residue treated once or twice with 1 to 2 c.c. of strong hydrochloric acid until the nitrate has been completely decomposed. The manganese is then precipitated by the addition of sodium carbonate, and the filtrate is evaporated and heated with zinc chloride, as described above.

B. B.

Comparison of the Kjeldahl-Wilfarth and Stock Methods of determining Nitrogen. E. Cavazzani and A. Cecconi. (*Ann. di Chim. e di Farmakol.*, 1894, xx., 87; through *Chem. Zeit.*)—In carrying out Stock's method (*THE ANALYST*, xvii., p. 109) for determining nitrogen, in which manganese dioxide is added to the sulphuric acid used to attack the substance, the nitrogen of which is to be determined, considerable frothing takes place in the early stages of the process, and care needs to be taken to prevent the liquid being projected from the flask. The end of the reaction can be recognised by the appearance of a green colour in the solution. Comparative analysis by the authors on blood, milk, and urine show that Stock's method is considerably quicker than the Kjeldahl process, but that it yields lower results. The differences are smallest with milk, and more noticeable with blood and urine, amounting to 8–9 per cent. The authors conclude that Stock's process is only available when rapidity of working is more important than extreme accuracy.

B. B.

Colour-reaction for the Detection of Sugars. E. Fischer and W. L. Jennings. (*Ber.* xxvii., 1355-1362.)—Some time ago Professor Emil Fischer showed that when certain sugars were dissolved in an alcohol belonging to the fatty series, and the solution saturated with gaseous HCl, crystalline compounds of the sugar and the alcohol, resembling in some respects the natural glucosides, are formed (see *Ber.* xxvi., 2401). The monohydric phenols appear to be incapable of furnishing such compounds with the sugar, but the polyhydric phenols yield amorphous colourless compounds, which seem also to be analogous to the natural glucosides.

Particularly interesting are the compounds of resorcinol with the aldoses, *i.e.*, sugars containing the group CHO, inasmuch as when these derivatives are oxidized in alkaline solution with such oxidizing agents as lead peroxide, mercuric oxide, and silver oxide, beautiful magenta colorations are produced. One of the most beautiful of these reactions, and one which serves for the detection of all aldoses (or of carbohydrates, which yield aldoses on hydrolysis with acid), is that obtained by warming the new compounds with Fehling's solution. It is so sensitive that in the case of arabinose-resorcinol this compound can be detected in a dilution of 1 : 50,000. The method is carried out as follows : To about 2 c.c. of the dilute aqueous solution of the substance to be tested 0.2 gramme of resorcinol is added, and the mixture saturated with HCl at 0° C. When a large amount of carbohydrate is present the test is allowed to remain at the ordinary temperature for an hour only, but when the quantity of carbohydrate is small it must be put aside for twelve hours. Subsequently the liquid is diluted, an excess of caustic soda added, and then warmed with Fehling's solution (only a few drops of the latter are needful when the quantity of carbohydrate is small). A reddish-violet coloration ensues, which is very characteristic ; it is fugitive after a time if the solution is strongly diluted. For the detection of insoluble carbohydrates, such as starch, these are triturated with water, and after adding resorcinol the mixture is saturated with HCl, as already described. The reaction has been proved with saccharose, lactose, maltose, dextrin, gum, glycogen, starch, and cotton-wool, besides the simpler sugars—(pentoses and hexoses). Normal urine exhibits the reaction very markedly.

The test is in many respects similar to that described by Molisch (*Monatsh. Chem.* vii., 198), but is not quite so delicate, and somewhat less convenient ; the authors are of opinion, however, that the test may be serviceable in confirming the indications obtained by Molisch's method.

A. R. L.

EXTRACTS FROM THE EVIDENCE GIVEN BEFORE THE SELECT
COMMITTEE ON FOOD PRODUCTS ADULTERATION, ON
JULY 11, 18 AND 25.

MR. RICHARD BANNISTER.

(Continued from page 215.)

3. EVIDENCE REFERRING TO MILK STANDARDS.

600. You mentioned the standard ; have you anything to say about limits ?—We went carefully over the whole ground. We have made experiments in the meantime and have always dried to constant weight, and we considered that 8.5 was the point where, if a milk came before us, it was necessary for the vendors to give some explanation about how it was that the milk was so low in solids-not-fat.

601. That is the line at which you begin to be suspicious, as it were, of water, and require a proof to be given that there is no water ?—Yes, exactly. There is a form of adulteration of milk that is rather common at the present time, and it is a very dangerous one, that is, the addition of separated milk to new milk. There is no

difficulty there about the solids-not-fat, because the solids-not-fat are increased; you will find that the fat will diminish, but the solids-not-fat will increase. If you find a sample of milk that will contain perhaps from 9 to 9·2 per cent. of solids-not-fat, and fat about 2·7 or 2·8, it is pretty certain that separate milk has been added to that sample.

(NOTE.—Refer to Dr. Bell's book for numerous instances of such milks included in his analyses of "genuine" milk.)

602. You think that that is a common method of adulteration now?—I think there is no doubt about it.

1540. What would you make your standard for solids-not-fat and fat?—We make it 8·5 for solids-not-fat and 2·75 for fat.

1728. You have altered your standard recently, have you not?—*We altered the limit from 2·5 to 2·75.*

1729. What was your standard before you altered it?—2·5.

1731. And 8·5 of solids-not-fat?—Yes.

1742. Has the Society of Public Analysts the same standard as Somerset House, within your knowledge?—*My opinion, from what I know from Public Analysts who have consulted us, and from the result of examination, is that the standard of the Society of Public Analysts seems to be 8·5 of solids-not-fat and 2·5 of fat.*

1743. Not three?—*Not three.*

1744. I am afraid that the Society of Public Analysts would rather not accept that statement, because, as I understand and am advised, their figures are 8·5 of solids-not-fat and 3 per cent. of fat.—*I have not seen any authority for the 3 per cent. of fat.*

1745. You have authority for the 2·5 per cent?—Yes, public analysts at starting made the standard 2·5 of fat.

(NOTE.—See verbatim report of Milk-Committee, ANALYST, vol. x., p. 216.)

1746. Do you not think it would be better if Somerset House and the public analysts could come to some common agreement as to the standard on which they could proceed to make analyses?—Really, so far as public analysts and Somerset House are concerned, the very few variations that there are between Somerset House and the public analysts show conclusively that the method of analysis now pursued is a very good method.

1734. Was it the fear of condemning milk that was pure that has induced Somerset House to keep their standard fairly low? I believe it is somewhat low as compared with what is adopted in other countries, and also by the Society of Public Analysts?—No; I think the whole subject depends upon the Act of Parliament. In other countries you will find that the Act of Parliament is different, and therefore they can go on a different line.

1735. Do I rightly understand you to suggest that the Act of Parliament defines the standard?—No, I do not; but the information that was obtained by the Committee on whose report the Act of Parliament was founded laid it down distinctly that all these little differences that you find in the feeding and keeping of cows, their housing, and different seasons, and all that, had to be taken into consideration.

1736. Then your standard is practically intended to represent a fair average analysis of milks obtained from herds, not from individual cows, because the milk of commerce is admitted, I believe, to be the milk of herds, milk in bulk?—Yes; but the difficulty of interpreting the Act of Parliament in that way arises from the fact that it applies to England, Scotland, and Ireland; and there is no doubt that so far as the large towns are concerned that is the fact; but there is a large quantity of single cow milk distributed in country places, and, therefore, if we had that definition it would follow as a matter of course that a person in a country place might be convicted when he was selling genuine milk.

1737. As a matter of fact, then, you fixed your border-line so as not to exclude single cow milk that might be poor?—But there are a great many single cows that come far below 8·5.

1738. In solids-not-fat?—Yes.

1739. What about the fats, too?—Some of the fats are lower.

1740. Lower than 2·75?—Yes, considerably.

1741. Would you condemn those milks?—No, I should not. If I knew the origin of the milks I should not condemn them; it is simply because we do not know the origin of the milk that we are compelled to put in the limit.

1750. With regard to fixing a standard, you are adverse to a hard and fast standard being fixed?—I am.

1635. I think Dr. Vieth has placed on record his analysis of over 120,000 samples of milk?—But, then, Dr. Vieth's experiments, as far as numbers are concerned, are of little value, because he was analyst to the Aylesbury Dairy Company, and, therefore, the examination of those samples of milk was done really in the examination of samples that were offered from the farmers from the dairies.

1636. Do you suggest that in consequence of his official connection with the Aylesbury Dairy Company those analyses were not an honest record?—No, I do not assume that for a moment; but what I mean to say is, that there is a contract entered into by all those dairy companies as to what quantity of solids-not-fat milk shall contain, and what quantity of fat, and it is the analyst's duty to see that the milk that they get comes up to their standard quality.

1637. But the fact remains that he analysed those 120,000 samples of milk?—*So far as the solids-not-fat and fat were concerned.* Of course, in the analysis of a sample of milk, as a rule, an analyst would go more fully into the analysis of that sample than is necessary for a commercial purpose of that kind.

719. Is sugar of milk ever added as an adulterant, or do you attach any importance to its estimation?—We never do estimate it in the analysis of a sample; it goes in the solids-not-fat.

720. I noticed also that in giving the results of analysis you mentioned the casein and albumen together; why was that?—*The only two points that we give in an ordinary analysis of milk are the solids-not-fat and the fat, and all those come in the solids-not-fat.*

1664. With regard to the standards that you may have at Somerset House, the information as to those has been disclosed, I believe, during the hearing of cases in which Somerset House has given evidence?—Yes, the limits.

1665. That is the way in which that information has become public, as a rule ; you told us that you had lately altered your standard of milk?—We have altered the limit of fat from 2.5 to 2.75.

1666. Would that be a matter that the public analysts would be acquainted with, or, rather, informed about?—We stated that distinctly to the different analysts who came up to see us. We have not written officially to the Society of Public Analysts.

4. EVIDENCE REFERRING TO DECOMPOSED MILK.

1667. With regard to your system of analysing milk, I understand that you have been in the habit of making allowances for decomposition, time-allowances?—We are compelled to make allowance for change, because in the samples of milk that you are speaking of they are old milks, not new milks.

1668. They would naturally be old milks before they arrive at Somerset House?—They must be.

1669. And in order to allow for the decomposition that has taken place you have a method of calculation?—We see what the change consists of, and work it back into the solids-not-fat.

1670. Would the public analyst know that table of allowances?—*It does not affect them in any way*, because they examine the samples of milk when they are fresh.

1671. Do you think that the table of allowances, the time allowances, that you have set forth for your own use, would give the same analytical results when you were dealing with the sample, as were apparent to the public analysts when they were examining the sample in a fresh condition?—Generally, but in special circumstances they would not ; and, therefore, we have abandoned the time allowance, and we try to trace up the change in the composition of the sample, and work it back again into solids-not-fat.

1672. I understand that you have rather altered your time allowances in that direction for the last year or two?—I do not think we have ; *practically they are the same*.

1673. When these milks come to you in a stale condition, are you obliged to state whether the article analysed is stale ; because under the Act the public analysts have to state in their certificate whether the sample is stale, or has been kept too long?—We examine whether it is fit for analysis, or whether there is any change in the composition of the sample that prevents an analysis being made. That is what I think the analysts have to state.

1674. Do you state that on your certificate?—*We do not state it on our certificate*, but if we come across a sample of milk and we find that it has been *improperly kept*, so that it is mouldy or decayed, we do not examine it. We say that it is not in a fit condition for examination.

1675. In how many cases, can you give us an idea, in recent years have you been unable to examine such a sample?—I should think in five or six cases.

1676. Does milk becoming decomposed alter its component parts, so far as solids are concerned?—Yes, the solids-not-fat are altered ; the fat remains practically the same.

1677. And it is by this principle of adjustment that you are able to arrive at the correct analysis?—Yes.

1678. May not some of these milks fail to give the same results?—They will not give the same results. If you take two samples of reference milk the results obtained will not be exactly the same, because they have been kept under different conditions; but we will take out the different substances that have been formed, and work them back again into solids-not-fat, so that if there is a quantity of alcohol formed in the one, and in the other there is none, the alcohol would be worked back again into solids-not-fat.

1679. Are you aware as to the conditions under which the samples have been kept before they were submitted to you?—We can generally tell from the analysis of samples under what conditions they have been kept.

1680. But when fermentation takes place, is there not a loss of substance incurred?—Yes; but there is a great deal of substance required to produce a certain quantity of alcohol, and when you have that quantity of alcohol you put it back again into the term of the substance that has been used up in the production of it.

1681. So that you can account by your system for loss of substance caused by fermentation?—We have found it to be so.

1682. You do not think that any of the conclusions which you arrive at by that process would be such as would cause you to give a decision contrary to that arrived at by public analysts?—Practically, *if the former analyses have been correctly made, they would agree with ours.*

1683. And there is no chance, you think, of any injustice being done to a public analyst by reason of this system which you adopt in dealing with milks that must necessarily be stale, many of which are in a state of fermentation before you examine them?—*I consider that there is no risk of injustice.*

1684. Do you ever test these milks for the presence of preservatives?—Yes.

1685. Would a milk bearing preservatives show a different result from a milk that was not preserved?—Yes, one would decompose more than the other.

1686. And when you detect preservatives, do you adopt a different procedure in coming to your final decision?—No; we take the results of the analysis, the quantity of alcohol formed, of ammonia, of acetic acid, and so on, and turn it back again into solids-not-fat.

1687. I refer to the time allowance?—I stated distinctly just now that we had abandoned the time allowance, because we find that in certain conditions the present system is better.

1688. I did not catch that; how long has that time allowance been abandoned, quite recently?—For the last two or three years, but *practically they are the same.*

1689. What are the same?—The two systems; if you take the time allowance and take our present method of examination; only no doubt our present system is the better system from a chemical point of view, because we take the determination of the different substances in the milk, and the changes that have taken place, and work them back again into solids-not-fat, so that it does not depend upon a time allowance.

1690. But in the communication which was referred to by Sir Charles Cameron, from the public analysts to Somerset House, was there not strong exception taken to that system of time allowance, to the system that you had?—I cannot tell all the details that were touched upon in that statement.

1692. Can you tell us what is the method that you adopt now since you abandoned this time allowance, so that the committee may understand how it is that you proceed in the case of stale samples?—Yes; the two substances that would change by keeping are the sugar and the casein. The sugar might be converted into lactic acid, in which there is no change in the solids-not-fat; or a portion of it might be converted into alcohol, a portion of the alcohol might be converted into acetic acid. As regards the curdy matter or casein, a small quantity of that might be converted into ammonia; accordingly we estimate the amount of ammonia; we estimate the amount of acetic or any other acid that may be present; we estimate the amount of alcohol, and then, taking these different estimations, we turn them back again into the amount of solids-not-fat.

1693. In preparing your certificate when you are called in as a court of appeal, as it were, do you certify whether or not any change has taken place in the nature of the article which might tend to interfere with the analysis?—If we consider that any change has taken place in the nature of the article which interferes with the analysis, we should not examine it.

1694. That would come under those samples which you refuse to analyse?—Yes.

1695. Under the Sale of Food and Drugs Act, are you asked for any opinion or to pronounce a judgment on the samples referred to you?—The reference to us under the Sale of Food and Drugs Act is the charge that is made before the magistrate; we have to examine the sample to see whether that charge has been properly made or not.

1696. Do you give a judgment on the charge, as it were, or do you simply express an opinion on the analysis?—We have to certify that in our opinion the analyses give certain results.

1697. I think you are frequently in the habit of using on your certificate these words as regards milk: "We are unable to affirm that water has been added"?—We have not used that form for years.

1698. I presume that the magistrate on seeing those words would rather feel inclined to think that there was disagreement between the public analyst and Somerset House?—*That really would be a misunderstanding, because the expression "unable to affirm" is only that you cannot tell from an examination of the sample whether water has been added or not.*

1699. On your certificate, do you give such information as will enable a magistrate to know whether the public analyst's certificate and yours are at variance on a matter of opinion or on a matter of fact?—*On a matter of fact.*

(Compare reply to questions 556, 557, and 558, September number of ANALYST, p. 212. Mr. Bannister said: I think that the difference is rather more in the interpretation of the results than to actual mistakes made. *We differed in the conclusions.*)

1700. I see that you say that your Department had referred to them since the beginning of the working of the Act 411 samples of milk, and that in 311 of these

cases you confirmed the public analyst, and in 96 you disagreed; 311 and 96 cases total 407; consequently there were four samples of milk in regard to which you neither disagreed with nor confirmed the analyst.—I think there must be some omission there or some error. I think there is nothing either one way or the other to come to the conclusion that we did not either agree or disagree with the analyst. I think there is some error in the figures.

(*To be continued.*)

CORRESPONDENCE.

SOMERSET HOUSE AND PUBLIC ANALYSTS.

[*The following correspondence has been received by the Editorial Committee from Mr. Allen with a request for its publication :*]

67, Surrey Street, Sheffield,
July 21, 1894.

DEAR MR. BANNISTER,—I was much surprised at the evidence you gave on Wednesday last before the Committee on Food Products Adulteration respecting a sample of malt-vinegar, marked "V.1," recently referred to Somerset House by the Sheffield City Magistrates. I have waited until I had the opportunity of seeing your evidence in print before communicating with you, as I thought I must have misunderstood you.

From your replies on page 93 of the evidence, it would almost seem as if you supposed that I had certified to the sample containing 80 per cent. of *acetic acid from wood*. On reference to the letter sent at your request by the Clerk to the Magistrates, you will find that what I alleged was that the sample contained 80 per cent. of *acetic acid derived from other sources than malt*, and only 20 per cent. derived from malted or unmalted grain. It was quite evident from the analysis that a part at least of this adulteration was not a fermentation-product, and therefore, in the face of the decision of the Birmingham Recorder, it would not have been correct to have called it 80 per cent. of *vinegar* from other sources than malt; but it is quite clear that the term "*acetic acid from other sources than malt*" includes sugar vinegar, and it has been a matter of great surprise to those concerned in the case that the referees did not report to this effect. Nothing was mentioned about wood-acid in the original certificate, or in the letter you received from the Clerk of the Magistrates, and I still fail to understand why you imported that element into the case.

I note that you say there is no test for wood-acid, and yet you certified to the presence of it, and shall be obliged if you will explain to me how you arrived at that conclusion.

I shall be glad if you will state, for the information of myself and the Health Committee of the City of Sheffield, how much, in your opinion, of malt-vinegar—that is, vinegar derived from malted and unmalted grain—the sample in question contained, as that was clearly the question referred to you. I alleged 20 per cent., and from the figures given by the defendant in the witness-box, the calculated amount closely corresponds with this result.

I do not know what the Inland Revenue officer was told when by your instructions he visited the defendant's works; but I have good reason to believe that the acetic acid employed by the defendant was that of the character commercially known as "1—8 acid," specially sold for adulterating vinegar, and containing a notable quantity of alcohol added for the express purpose of deceiving the referees and public analysts. You appear to have fallen into the trap, and supposed it to be a fermentation-product.

I learn from your evidence that you are always ready to communicate information to

public analysts when requested, and shall therefore be obliged if you will let me have a reply to the foregoing questions at your early convenience.—I remain, yours very truly,

RICHARD BANNISTER, Esq.,

(Signed)

ALFRED H. ALLEN.

Inland Revenue Laboratory,

Somerset House, London, W.C.

The Government Laboratory, Somerset House, W.C.,

July 27, 1894.

DEAR SIR,—I am sorry that, through pressure of work connected with the Committee on Food Products Adulteration, I have been compelled to postpone my reply to your letter till now.

The reply that I gave to Mr. Kearley's question arose from his quotation of the reference letter of Mr. Charles E. Vickers, Clerk to the Sheffield justices, which was that the nature of the alleged adulteration consisted of 20 per cent. of malt-grain vinegar, and 80 per cent. of *diluted* acetic acid not derived from malted or unmalted grain.

In your letter you leave out the word *diluted*, which qualifying word justified me in concluding that the origin of the 80 per cent. of diluted acetic acid was strong acetic acid, which had been diluted to the required strength.—Believe me, yours very truly,

A. H. ALLEN, Esq.,

(Signed)

R. BANNISTER.

Public Analyst, Sheffield.

67, Surrey Street, Sheffield,

July 28, 1894.

DEAR SIR,—I am in correspondence with Mr. Bannister, the Deputy-Principal of the Somerset House Laboratory, respecting the certificate he gave on a sample of vinegar recently referred to him by the Sheffield city magistrates.

Mr. Bannister says he misunderstood the nature of my certificate, being misled by the words of your letter of instruction. May I ask you to oblige me with a copy of your letter to Somerset House in reply to their request for information as to the nature of the alleged adulteration.

Thanking you in anticipation,—I am, dear sir, yours truly,

CHARLES E. VICKERS, Esq.,

(Signed)

ALFRED H. ALLEN.

Clerk to the Magistrates, Sheffield.

The Court House, Sheffield,

July 30, 1894.

DEAR SIR,

VINEGAR.

Your letter of the 28th inst. to hand, and, as therein requested, forward you a copy of my letter to Mr. Bannister respecting the above.—Yours truly,

ALFRED H. ALLEN, Esq.,

CHAS. E. VICKERS,

PER G. H.

67, Surrey Street, Sheffield.

The Court House, Sheffield,

June 5, 1894.

SIR,

THE SALE OF FOOD AND DRUGS ACT, 1875.

Your letter and enclosure—extract from the Local Government Board's circular of September 30, 1875—received, and in reply beg to say that the nature of the alleged adulteration consisted of 20 per cent. of malt-grain vinegar, and 80 per cent. of diluted acetic acid not derived from malted or unmalted grain.—I am, sir, your obedient servant,

CHAS. E. VICKERS (PER G. H.),

Clerk to City Justices.

R. BANNISTER, Esq.,

Inland Revenue Laboratory, Somerset House, London, W.C.

67, Surrey Street, Sheffield,
August 3, 1894.

DEAR SIR,—I enclose you a copy of the certificate which I gave on the sample of vinegar recently referred to you, which you will see differs materially from the description of the accusation given in the letter of instructions of the Clerk to the Magistrates. I quite agree with you that the word "diluted" bears a different signification from "dilute," and justified your misapprehension of the real nature of the accusation. It is a great pity that Mr. Vickers did not send you an actual copy of my certificate.

I note from your evidence that you are always willing to communicate your limits to Public Analysts who apply for them, and I shall therefore be glad if you will inform me what limits of composition you adopt for malt-vinegar. May I also trouble you to state the limits you adopt for tincture of rhubarb, as this is an article which one of my inspectors not unfrequently sends me.

Thanking you in anticipation for an early reply,—Believe me, yours very truly,
R. BANNISTER, Esq., (Signed) ALFRED H. ALLEN.
Somerset House Laboratory, London, W.C.

ANALYST'S CERTIFICATE.

TO THE HEALTH COMMITTEE OF THE BOROUGH OF SHEFFIELD,—I, the undersigned, Public Analyst for the Borough of Sheffield, do hereby certify that I received on April 2, 1894, from Inspector Harrison a sample of "malt-vinegar" for analysis, and was marked "V.1," and have analysed the same, and declare the result of my analysis to be as follows. I am of opinion that the said sample contained the parts as under :

Malt or Grain Vinegar	20 per cent.
Dilute Acetic Acid not derived from Malted or Unmalted Grain	80 " "

100

As witness my hand this 18th day of April, 1894.

(Signed) ALFRED H. ALLEN,
At Sheffield.

Government Laboratory, Somerset House, W.C.
August 4, 1894.

DEAR SIR,—In reply to your letter of yesterday, asking for the limits we adopt for Malt-Vinegar and Tincture of Rhubarb, I beg to inform you that we are unable to adopt a limit in either case, and are compelled to make each sample submitted to us stand on its own merits. The difficulties connected with these examinations are, I am sure, well known to you, and you will fully appreciate the difficulty of speaking more specially on the subject.—Believe me, yours very truly,

ALFRED H. ALLEN, Esq. (Signed) R. BANNISTER.

67, Surrey Street, Sheffield.
August 14, 1894.

DEAR SIR,—I duly received, and am much obliged for, your letter of the 4th inst., and to which I should have replied sooner but for the intervention of the holidays.

I fully appreciate the difficulty of laying down limits for such articles as malt-vinegar and tincture of rhubarb, but unfortunately in practice one is constantly required to meet this difficulty. I not infrequently have to report on samples of tincture of rhubarb, which I do to my own satisfaction on the basis of a series of experiments instituted for the purpose ; but I have no certainty that, in the event of my analysis being disputed, and the sample referred to

Somerset House, your opinion will not be formed on wholly different data. I therefore appeal to you to inform me what data and limits of composition you would rely on supposing that to-morrow a sample of tincture of rhubarb were referred to you under the Act.

Similarly, I request you to inform me what data and limits of composition you would rely on in the case of a sample of malt-vinegar referred to you. I am anxious for this information in order to avoid the annoyance and scandal which result from the disagreement of the Referees with the certificate of a Public Analyst. As an instance in point, I may say that a sample of vinegar, which the manufacturers admitted to me to be largely adulterated with acetic acid, was declared by you to be genuine malt-vinegar.

While urgently requesting a reply on the general questions above submitted, I shall be greatly obliged if you will give me your opinion on the following figures, which I obtained by the analysis of a sample sold as "malt-vinegar." The case is quite distinct from the one above-mentioned, where you passed as genuine one admittedly largely adulterated.

Specific gravity	1.0130
Per 100 parts of vinegar:—							
Acetic acid	4.56
Total extractive matter	1.70
Ash	0.34
Alkalinity of ash in terms of K_2O	0.056
Phosphoric acid (P_2O_5)	0.023
Sulphuric acid (SO_3) combined	0.020
Nitrogen	0.029

Requesting the favour of a reply at your early convenience,—Believe me, yours very truly,

R. BANNISTER, Esq.,

(Signed)

ALFRED H. ALLEN.

Government Laboratory,

Somerset House, London, W.C.

Laboratory, Somerset House, London.

August 15, 1894.

DEAR SIR,—With reference to the fixing of Standards for tincture of rhubarb and malt-vinegar, I have nothing to add to the remarks contained in my previous letter. It is practically impossible to lay down general limits for such articles, and I think the experience of the last six months, with reference to the composition of malt-vinegar, must prove how imprudent it has been for analysts to rely on so-called standards, which before the Magistrates have been proved to have no solid foundation.

May I trespass on your kindness for such information about the sample of vinegar you refer to, which was passed by us as genuine malt-vinegar, and which admittedly was largely adulterated, as will enable me to identify it. I shall be much obliged if you will do so.—Yours very truly,

ALFRED H. ALLEN, Esq.,

(Signed)

R. BANNISTER.

Public Analysts' Laboratory,

67, Surrey Street, Sheffield.

67, Surrey Street, Sheffield.

August 16, 1894.

DEAR SIR,—I beg to acknowledge your letter of August 15, stating that it is practically impossible to lay down standards for the composition of tincture of rhubarb and malt-vinegar, and that you consider the experience of the last six months with reference to the composition of malt-vinegar proves how imprudent it has been for analysts to rely on so-called standards. I

regret you do not see your way to give me any assistance even in a specific case like that in which I sent you the figures.

I am sorry I must decline to give you the information you request respecting the sample of adulterated vinegar which you certified to be genuine. The manufacturers consulted me when their customer was summoned, and admitted that there was a very large percentage of added acetic acid present. Under the circumstances I declined to assist them in their defence; but they succeeded in having the case referred to Somerset House, and the Referees reported it to be genuine malt-vinegar, much, of course, to the elation of the manufacturers. You will see that I cannot give you the information which will enable you to identify the vinegar without making known to you the name of the firm who consulted me, and as they sought my advice in confidence, I am bound to preserve their secret.—Yours very truly,

R. BANNISTER, Esq.,

(Signed) ALFRED H. ALLEN.

Government Laboratory,

Somerset House, London, W.C.

Laboratory, Somerset House, London.

August 17, 1894.

DEAR SIR,—I am very much surprised that you decline to give me the information I ask for to identify the sample of vinegar you allege we certified as malt-vinegar, when it contained a "very large percentage of added acetic acid," because I have seen the same statement made in a class paper.

If, therefore, the information could, without any breach of confidence to your client, be supplied to a newspaper, I think that common justice, as well as professional etiquette, demands that we should be told what sample it was to which you refer, to enable us to clear up the charge you bring against us.—Yours very truly,

ALFRED H. ALLEN, Esq.,

(Signed) R. BANNISTER.

Public Analysts' Laboratory,

67, Surrey Street, Sheffield.

67, Surrey Street, Sheffield.

August 18, 1894.

DEAR SIR,—I am duly in receipt of your letter of yesterday referring to the sample certified by you to be pure malt-vinegar, but which I was informed by the manufacturers contained a large percentage of added acetic acid. I am not surprised that you have seen the statement made in a "class paper," and notice that *Food and Sanitation* of to-day contains a reference to the matter. I have made no secret of the fact among my brother Analysts and the Authorities for whom I act, and as the Editor of *Food and Sanitation* is very enterprising, it is not astonishing that the information has reached him; but I did not communicate it, nor have I ever contributed to that journal, except such letters as have appeared over my signature.

I think you will see on reflection that it is a very different thing to make freely known the fact that you missed the adulteration, and to do as you desire, which is practically to inform you of the name of the manufacturer. Everyone has his own standard of ethics, and I have no desire to dictate to you on such a subject; but it is not in accordance with my idea of professional etiquette to make known the name of a client who consulted me confidentially on a matter respecting which he was well aware he was a transgressor.

I may remind you that you have not complied with my request for your opinion respecting

certain data obtained by the analysis of a sample of vinegar submitted to me.—Believe me,
yours very truly,

R. BANNISTER, Esq.,
Government Laboratory,
Somerset House, London, W.C.

(Signed) ALFRED H. ALLEN.

Laboratory, Somerset House, London, W.C.

August 20, 1894.

DEAR SIR,—I beg to acknowledge the receipt of your letter of the 18th inst., and am certainly very much surprised to learn therefrom that your idea of professional etiquette permits you to put in circulation a statement respecting the analysis of a sample which we are said to report as "pure malt-vinegar," and when I ask you to supply me with the information necessary to prove the truth or otherwise of the statement, you decline to give it.

Happily such a standard of ethics is rare, even in these days of systematic misrepresentation.

My letter of August 4 gives you the reason why I declined to give an opinion on the data you submitted to me. There are many points to be considered in the analysis of a sample of vinegar, in addition to the figures you forwarded.—Yours very truly,

ALFRED H. ALLEN, Esq.,
Public Analysts' Laboratory,
67, Surrey Street, Sheffield.

(Signed) R. BANNISTER.

67, Surrey Street, Sheffield.

August 23, 1894.

DEAR SIR,—I am duly in receipt of your letter of the 20th inst.

Every official action of the gentlemen acting as Referees under the Sale of Food and Drugs Act is naturally of great interest and importance to Public Analysts, and fair subject for comment. Hence I feel bound, in the cause of truth and the interests of my brother Analysts, to make them acquainted with your failure to report the presence of added acetic acid in the vinegar in question; and the more so in the face of your professed inability to give any indication whatever of the standards or limits you have adopted or would adopt in judging of any similar sample referred to Somerset House.

It is so clearly for the public benefit that Analysts should know that their reports on similarly adulterated samples are likely to be contradicted on appeal, that I am surprised you should feel any dissatisfaction with my taking any steps to attain so desirable an object.

I note your implied doubt of the accuracy of my statement that you reported favourably on a sample sold as malt-vinegar which the manufacturers admitted to contain a large percentage of added acetic acid. I regret this, but it is evident that, if my assurance does not carry conviction, nothing short of an application by you to the manufacturer in question would enable you to "prove the truth of the statement." I have already explained the circumstances under which the information came to me, and can only regret that you should suppose it possible to induce me to commit such a flagrant breach of confidence by making the insinuations contained in your letter.—Yours very truly,

RICHARD BANNISTER, Esq.,
Government Laboratory,
Somerset House, London, W.C.

(Signed) ALFRED H. ALLEN.

Laboratory, Somerset House, London, W.C.

August 24, 1894.

DEAR SIR,—Your letter of the 23rd inst. has come duly to hand ; and, in acknowledging it, I must state that I should have been quite content if you had, by inquiry, shown the same anxiety to verify the accuracy of the statement made by your client as you have been in keeping back any information which could lead to the identification of the sample described in one of your letters as genuine malt-vinegar, and in another as pure malt-vinegar, neither of which expressions having, to my knowledge, been used in any of our certificates.

This inquiry you have not made, and you make your case worse by giving, as a reason for circulating the report, my "professed inability to give any indication whatever of the standards or limits you have adopted, or would adopt, in judging of any similar sample referred to Somerset House," when you are quite aware you circulated the tale long before my letter of the 4th inst. was written.

I must refer you again to my letters of the 4th and 15th inst. for the reasons I gave for my inability to define a limit in such cases.

I have never wished, or asked, you to commit any breach of confidence, but I naturally expected that you would, as an act of simple justice, be prepared to substantiate any charge you might make against this department ; but I regret to find that this is not the case.—Yours very truly,

ALFRED H. ALLEN, Esq.,
67, Surrey Street, Sheffield.

(Signed) R. BANNISTER.

67, Surrey Street, Sheffield.

August 28, 1894.

DEAR SIR,—A sample of ground ginger was recently referred to you by the magistrates sitting at Bingley Petty Sessions. In this case you reported the presence of 8·82 per cent. of mineral matter, of which 2·11 per cent. was sand, and stated that this would be excessive if found in high-class ginger which had been washed before being ground, but the results obtained were not greater than were sometimes found in low-priced ginger which had been ground as imported. Will you kindly inform me of the maximum limit of total ash and sand you would permit in a sample of ground ginger of unknown origin and unknown nature, and in the absence of any information whether it was ground before or after being washed ? I shall be further obliged if you will inform me whether you took any steps in the case in question, and, if so, what, to ascertain whether, as a fact, the ginger named had or had not been washed before being ground.

According to a printed account before me of a case recently heard at Wood Green, in which a sample of milk had been referred to Somerset House for analysis, Mr. Helm and yourself are reported to have certified to the presence of 8·25 of solids-not-fat, and 2·62 of fat, at the same time expressing such an opinion as led the magistrates to dismiss the case. I shall be glad to be informed whether the above report is substantially correct, and, if so, what are the limits you *actually* adopt for fat and solids-not-fat, as it would appear from the report in question that you do not in practice adhere to the statement made by you in evidence before the Select Committee on Food Products Adulteration that your limit for solids-not-fat was 8·5 per cent., and that you had recently raised your former standard of 2·5 of fat to 2·75.

The great importance of the practice of Somerset House must be my apology for troubling you in this matter.—Yours very truly,

R. BANNISTER, Esq.,
Government Laboratory,
Somerset House, London, W.C.

(Signed) ALFRED H. ALLEN.

67, Surrey Street, Sheffield.

August 29, 1894.

DEAR SIR,—I find your letter of August 24 awaiting me after a short absence from home.

I have already explained that I have it *on the authority of the manufacturer himself* that there was a large proportion of added acetic acid present in a sample of vinegar referred to you, and the case was dismissed in consequence of the omission from your certificate of any mention of this added acid, which, therefore, I presume your analysis failed to detect. I do not see how I could possibly have further verified "by inquiry" the accuracy of the statement made by my client as to the composition of the vinegar he himself had manufactured; but I may say that I had *previously detected the fact by analysis* of the vinegar, and it was when the manufacturer was informed of this that he admitted the accuracy of my conclusions.

I understand from your letter that you distinguish, and wish me to draw a distinction, between "genuine malt-vinegar" and "pure malt-vinegar" on the one hand, and "malt-vinegar" without either of these descriptive adjectives on the other. If your contention is that the term "malt-vinegar" simply is legitimately applicable to an article containing a large proportion of added acetic acid, it appears to me a pity that Public Analysts should not be made aware that you hold such a very unusual view of the nature of malt-vinegar.

But if you will have the goodness to peruse my letters again, I think you cannot fail to see that I used the term "genuine" and "pure" in contradistinction to *adulterated*, and did not imply that either of these words was actually used in your certificate on the vinegar in question. But, in order that there may be no misunderstanding, I repeat that a sample sold as "malt-vinegar" was certified by a public analyst to contain a large proportion of acetic acid from another source, which accusation was admitted by the manufacturer to be correct. On reference to Somerset House, you reported in terms which implied that the sample was of unexceptionable character, thereby leading to a dismissal of the summons and a deplorable failure of justice.

If you will kindly refer to my letter of August 18, you will see that I there state that I *had* made no secret among my brother Analysts and the authorities for whom I act of your faulty certificate in the above case, and I undoubtedly mentioned the matter at a date considerably prior to the commencement of this correspondence. But now, in the face of your professed inability to give any indication whatever of the standards or limits you have adopted, or would adopt, in judging of any similar sample submitted to you, I feel bound to make my brother Analysts *in general* acquainted with your failure to report the presence of added acetic acid in the vinegar in question. As pointed out in my last, it is clearly for the public benefit that Analysts should know that their reports on similarly adulterated samples are likely to be contradicted on appeal.

I strongly demur to your statement that I am not prepared to substantiate the charge I have made in this matter against your department. On the contrary, I am quite prepared to repeat on oath the statements contained in this and previous letters to you, and not improbably shall have an opportunity of doing so.

In your letter of August 20, you state that there are many points to be considered in the analysis of a sample of vinegar in addition to the data I gave in my letter of August 14. Will you please tell me what these points are, and I will, to the best of my ability, give you the further information you require in order to express an opinion on the sample? I fully agree with you that it is desirable to base an opinion on the fullest possible analytical and physical data; but I trust you will pardon my saying that I do not think the information obtained by an Inland Revenue officer as the result of his visit to a vinegar works should form the basis of a certificate given by the Somerset House chemists on a sample of vinegar

from the same works submitted to them *for analysis* under the Sale of Food and Drugs Act.—

Yours very truly,

R. BANNISTER, Esq.,

Government Laboratory,

Somerset House, London, W.C.

(Signed)

ALFRED H. ALLEN.

Laboratory, Somerset House, London, W.C.

August 30, 1894.

DEAR SIR,—I have duly received your letters dated the 28th and 29th inst. As I deny your assertion that a sample of vinegar referred here under the Food and Drugs Act was reported as malt vinegar when it contained added acetic acid, and as you are unable to supply any information which will enable me to identify the sample you refer to, I must decline to continue this correspondence.

I will simply add that at the proper time I shall be happy to show the Select Committee on Food Products Adulteration that in the Woodgreen milk case, to which you refer, our certificate of analysis is quite consistent with the evidence I have already given with regard to the composition of milk.—Yours very truly,

ALFRED H. ALLEN, Esq.,

Public Analyst's Laboratory,

67, Surrey Street, Sheffield.

(Signed)

R. BANNISTER.

67, Surrey Street, Sheffield.

August 31, 1894.

DEAR SIR,—I am duly in receipt of your letter of yesterday.

I am not concerned by your denial and implied disbelief of my statement respecting your erroneous vinegar certificate, as there happens to be a third person whose testimony is available in absolute confirmation of my charge. You will learn more about the matter in due time.

I must remind you that there are several points in my previous letters which you have omitted to notice, and for your convenience will re-state them :

1. Will you kindly inform me what information you require in addition to the data given in my letter of August 14th to enable you to express an opinion on the sample of vinegar therein referred to ?

2. Will you kindly state the maximum limits of total ash and of sand you would permit in a sample of ginger of unknown origin, and in the absence of any information whether it was ground before or after being washed ? Also please inform me whether you took any steps in the case of the sample recently referred to you by the Bingley magistrates to ascertain whether, as a fact, the ginger in question had been washed before being ground.

3. Will you please state what are the limits for fat and solids-not-fat in milk which you adopt in practice ?

In your evidence before the Select Committee on Food Products Adulteration you stated that you are always ready to communicate to a Public Analyst who may apply to you your limits or any other information in your power, and I shall accordingly esteem it a favour if you will let me have a reply on the above points at your earliest convenience.

Thanking you in anticipation, I am, yours very truly,

R. BANNISTER, Esq.,

Government Laboratory,

Somerset House, London, W.C.

(Signed)

ALFRED H. ALLEN.

Laboratory, Somerset House, London, W.C.

September 1, 1894.

DEAR SIR,—Your letter of the 31st ult. has been duly received, but for the reasons given in my letter of the 30th I must decline further correspondence —Yours very truly,

ALFRED H. ALLEN, Esq.,

(Signed) R. BANNISTER.

67, Surrey Street, Sheffield.

67, Surrey Street, Sheffield.

September 3, 1894.

DEAR SIR,—I am duly in receipt of your note of the 1st inst.

I regret that your failure to induce me—by aspersions, unworthy of you and unmerited by me—to commit a flagrant breach of confidence, is regarded by you as a proper and sufficient reason for refusing to afford me such information as you told the Select Committee on Food Products Adulteration you were ready to give to a Public Analyst who applied for it.—Yours very truly,

R. BANNISTER, Esq.,

(Signed) ALFRED H. ALLEN.

Government Laboratory,

Somerset House, London, W.C.

To the Editors of THE ANALYST.

2, Fisher Street, Red Lion Square, London, W.C.

September 22, 1894.

DEAR SIRS,—In Mr. Bevan's letter appearing in this month's copy of THE ANALYST, he has unknowingly not stated the exact facts of the case as far as my evidence was concerned. My analysis of the sample in question in duplicate was as follows :

Total Solids	11.43	Repeat. 11.47
Fat	2.82	2.86
Solids-not-fat	8.61	8.61

The report issued by my firm was "very poor milk, but no proof of adulteration." I supported our certificate at the hearing of the case, to the extent of stating that the milk "might be adulterated," or that it "might be a naturally very poor sample," and that "if the former, it was probably both skimmed and watered." It may also interest your readers to know that apart from the scientific evidence, proof was forthcoming that the milk had not been tampered with.

I must ask you in fairness to myself to publish the above facts, in correction of the mistaken view already put forward.—I am, yours faithfully,

A. J. DE HAILES.

To the Editors of THE ANALYST.

4, New Court, Lincoln's Inn, London, W.C.,

September 27, 1894.

DEAR SIRS,—Will you allow me to point out an error in my letter of August 30, published in the September number ?

The Total Solids should read 11.00, and not 11.10.

I should also like to mention that when Mr. De Hailes analysed the sample it was at least fourteen days old.—Yours faithfully,

EDWARD BEVAN.

THE ANALYST.

NOVEMBER, 1894.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on October 3rd at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner took the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed for election as members: E. A. Hancock, F.C.S., Government Laboratory, St. Kitts, West Indies (by the Council); Thomas Alex. Pooley, B.Sc. (London), F.I.C., 34, Old Broad Street, E.C.; John Clough Thresh, D.Sc. (London), M.B., B.Sc. (Victoria), D.P.H., Chelmsford, Essex; Robert Waterhouse, 101, Leadenhall Street, E.C.; William G. Wagner, 101, Leadenhall Street, E.C.

Mr. Bevan then read the following paper:

THE LOSS OF TOTAL SOLIDS IN MILK ON KEEPING.

SOME time ago a number of samples of milk arrived at my laboratory late on Saturday morning. The specific gravity of each was taken, and the fat estimated by the Leffmann-Beam process. Such as were suspected of being adulterated were weighed out into platinum dishes, and placed in desiccators until the following Monday morning, when they were evaporated to dryness in the usual way. My assistant, Mr. Merriman, called my attention to the fact that the total solids found in every case differed by about 1.0 per cent. from that calculated by the Richmond scale. As a rule, we find the concordance between the calculated and observed total solids to be very close, and we were at a loss to account for the discrepancy. The total solids were repeated on fresh portions of the milks, when they were found to correspond with the calculated quantities. Further analysis showed the milks to be perfectly normal as regards the relationship between gravity, fat, and total solids.

The discrepancy was so marked in every instance that we were forced to the conclusion that a loss of total solids had taken place on standing, and we immediately instituted a number of experiments with a view to showing if this really were the case. The following are the results obtained:

TABLE A.

	Total Solids.	Loss in Total Solids.
Evaporated immediately standing in open dish	... 11.78	
" after 24 hours	... 10.79	0.94
" after 48 hours	... 10.38	1.35
" after 120 hours	... 9.42	2.31

A large number of experiments were made showing similar results.

I communicated the results of my experiments to Mr. Richmond, who informed me that Dr. Veith had observed the same thing, but in every case his loss in total solids was much less than mine. Notwithstanding that his results are published in *THE ANALYST* for 1882, the phenomenon seems to have been unknown to several chemists with whom I discussed the matter; nor have I been able to discover any notice of the fact in any work treating of milk analysis, beyond the following statement in *Leffmann and Beam's* book on "The Analysis of Milk and Milk Products": "Richmond has pointed out that if the evaporation be slow, some decomposition occurs, and the residue will be brown, but that if the larger portion of the water be evaporated quickly, a white residue is obtained."

If such a loss as 0.94 occurs in twenty-four hours, it is certain that an appreciable loss may arise in a very few hours, especially in warm weather. The importance of the matter must be my excuse for reading this paper to-night.

In the experiments I have already recorded, the milks were weighed out in the fresh state and evaporated at certain intervals without neutralizing, so that the loss includes everything volatile at the time of evaporation. If immediately before evaporation the milk is neutralized with decinormal soda the loss is not so great, probably due to the fact that the lactic or other acid formed is completely retained by the soda. Lactic acid is stated in text-books to be non-volatile at 100°, and Dr. Bell in "The Chemistry of Foods," Part II., says (p. 15), "and as the acid is not volatile, its weight is correctly indicated on drying the milk."

Actual experiment in my laboratory shows that lactic acid is volatile. To 5 grammes of fresh milk I added 0.1445 pure lactic acid. The total solids obtained amounted to 0.6610 grammes, as against 0.5784 in the original milk. This corresponds to a loss of 42.8 per cent. of the lactic acid. By merely evaporating a weak solution of lactic acid in a platinum dish at 100° a considerable loss is sustained. I have, moreover, made a number of experiments showing the difference in the solids-not-fat in sour milk between neutralizing and not neutralizing :

TABLE B.

	Original Milk.	Kept 48 Hours.	Kept 96 Hours.
Total solids neutralized	13.30	13.25	13.00
" " not neutralized	13.30	13.12	12.70
Difference	0.03	0.13	0.30
Total acidity reckoned as lactic acid ...	0.23	0.93	1.05

The samples were preserved in well-corked bottles.

In the following table (C) will be found the results obtained after neutralizing, together with the acidity in the case of milks kept in open dishes :

TABLE C.

	Loss in total Solids.	Total Acid reckoned as Lactic.
	Neutralized	
Evaporated after standing in open dish } for 24 hours	0.30	0.220
Evaporated after 48 hours	0.65	0.630
" after 96 hours	0.88	0.594

It is important to notice that after standing ninety-six hours the loss in total solids amounts to 0·88 per cent., with a total acidity of 0·594 per cent. On comparing these numbers with those obtained with milks that have been kept in well-corked bottles (table B), it will be found that there is absolutely no connection between the loss and the percentage of acid formed. Moreover, it is remarkable that the acidity is greater in the milk kept for forty-eight hours than it is in that kept for ninety-six. It may be argued that a mistake has possibly been made either in the estimation of the total solids or in the acid; but numerous examples of similar "discrepancies" convince me at least that the differences are due to slight unknown variations in the conditions under which the samples have been kept. The following table shows the losses sustained by keeping milk under varying conditions, together with the amounts of total acid:

TABLE D.

Total Acidity reckoned as Lactic Acid.	Loss in total Solids (neutralized).	Conditions of keeping.	Composition of Original Milk.	
			Total Solids.	Fat.
1. 0·226	0·30	24 hours in open dish	12·38	3·50
2. 0·630	0·65	48 " "	"	"
3. 0·594	0·88	96 " "	"	"
4. 0·700	1·36	120 " "	"	"
5. 0·860	2·80	120 hours in open dish + 2% Na_2CO_3 ...	"	"
6. 0·50	1·16	120 hours in open dish + 2% salicylic acid ...	"	"
7. 0·774	2·17	120 hours in narrow beaker	"	"
8. 0·709	1·35	120 hours in flask with current of air passing ...	"	"
9. 1·04	0·74	51 days in corked bottle	9·20	0·29
10. 0·93	1·22	67 " "	12·00	3·28
11. 1·61	0·35	71 " "	11·50	3·54
12. 0·96	1·05	72 " "	11·51	3·18

The important point to be noticed is the disproportion between the total acidity and the loss in total solids.

If, then, there are such extraordinary differences in the ratio of the loss in total solids to the amount of total acidity (and the same thing would doubtless apply to the alcohol, ammonia, and acetic acid formed), one is inclined to ask what value, if any, is to be attached to a system of estimating these bodies in milks decomposed beyond recognition, and endeavouring to arrive at an opinion as to the original condition of the sample. Although reference has already been made in this journal, I cannot do better than repeat in this connection the questions put to Mr. Bannister and the answers he gave before the Select Committee:

1669. And in order to allow for decomposition that has taken place you have a method of calculation?—We see what the change consists of, and work it back again into solids-not-fat.

1678. May not some of these milks fail to give the same results?—They will not give the same results. If you take two samples of reference milk the results obtained will not be exactly the same, because they have been kept under different conditions;

but we take out the different substances that have been formed, and work them back again into solids-not-fat, so that if there is a quantity of alcohol formed in the one, and in the other there is none, the alcohol would be worked back again into solids-not-fat.

1682. You do not think that any of the conclusions which you arrive at by that process would be such as would cause you to give a decision contrary to that arrived at by public analysts?—Practically, if the former analyses have been correctly made, they would agree with ours.

1686. And when you detect preservatives, do you adopt a different procedure in coming to your final decision?—No; we take the results of the analysis, the quantity of alcohol formed, of ammonia, of acetic acid, and so on, and turn it back again into solids-not-fat.

1689. What are the same?—The two systems, if you take the time allowance, and take our present method of examination; only no doubt our present system is the better system from a chemical point of view, because we take the determination of the different substances in the milk, and the changes that have taken place, and work them back again into solids-not-fat, so that it does not depend upon a time allowance.

With regard to the determination of alcohol: As a rule, the samples referred to Somerset House are twenty-eight days old; now the old time allowance, which, as Mr. Bannister has stated, is practically the same as the new and, from a chemical point of view, better system, is, according to Dr. Bell, 0.48 per cent. I think it may be taken for granted that in no case does the weight of the sample exceed 6 ounces. Six ounces is equal to 168 grammes; 0.48 per cent. on 168 grammes is 0.806 gramme, which represents the total actual loss, and includes, of course, alcohol and carbonic acid.

Even if the whole of this loss of 0.806 gramme were alcohol, its accurate determination would be an exceedingly difficult matter. But when we consider that the total solids have also to be determined, presumably in duplicate, the difficulty is increased. But of course a considerable proportion of the loss is represented by carbonic acid, so that the amount of alcohol that can possibly be present would be very small. A number of experiments are in progress which I hope will throw light on the relationship between the loss in solids and the carbonic acid evolved.

DISCUSSION.

Mr. Hehner said that Mr. Bevan had given them a series of very interesting observations on the subject of the alterations which occurred in milk during keeping. Though this subject had been often discussed on previous occasions in that room, yet it was desirable that it should be approached by a new mind and from a different point of view. Mr. Bevan's observations when further expanded could not fail to be of considerable utility.

Mr. Richmond said that in a series of experiments which he had made some years since, he had found that lactic acid, especially in concentrated solution, was distinctly volatile when distilled with water. There was not nearly the same loss with the commercial syrupy acid, because it contained a considerable proportion of

anhydride, and it had to be boiled for some time before the anhydride was converted into the acid. With regard to the loss of total solids on keeping milk, taking Mr. Bevan's figures, it appeared, roughly speaking, the greater amount of acid formed, the less was the loss—a fact which his own experience confirmed. In some samples there was a large amount of acid formed and a moderate loss of total solids, but in one sample, which had been kept for six or seven weeks, though only a small amount of acid had been formed, there was a loss of 5 or 6 per cent. in the total solids. Mr. Stokes had published results of the same kind. In Dr. Bell's book it is stated that when there is an excessive amount of acid formed, a greater amount of decomposition was indicated, and presumably a greater loss of total solids. During the last three or four years there had appeared in the journal of the Chemical Society a series of papers by Dr. Percy Frankland on the fermentation of sugar with pure cultivations of different organisms; and it had been found that there was a great difference in the nature of the products according as the fermentation was conducted in an open or a closed vessel. In the former case the products were generally alcohol, acetic acid, and frequently other acids (succinic); in the latter, alcohol, acetic acid, and *formic acid*, while much hydrogen and carbon dioxide were evolved. The ethacetic fermentation was found to be very general, and the quantitative yields of alcohol, etc., varied within fairly wide limits. Dr. Frankland's researches indicate very strongly that in keeping milk in a bottle there was likely to be considerable difference in the products of decomposition according as the bottle was tightly corked or the reverse. The products of the decomposition of milk were so varied that it was impossible to fix any time allowance. The estimation and calculation back of the alcohol and other bodies formed seemed to be of doubtful accuracy. He had found in the case of koumiss, which was milk fermented by the action of various organisms under constant conditions, that it was possible to find an approximately constant factor, though hardly good enough for analytical purposes; but it is extremely doubtful if this can be done when milk is subjected to the action of all kinds of organisms. In reply to Mr. Bevan, Mr. Richmond said that koumiss was not fermented by a pure culture of bacteria, but by a special cultivation in which several (at least three) species of organisms were present.

Mr. Allen said that at the present time he had a series of experiments in progress with a view of learning more about the rate of change of milk by keeping. A number of tubes were taken, and 10 grammes weight of a sample of new milk placed in each. The tubes were then hermetically sealed, and preserved at the ordinary temperature. Every week one of the tubes was opened, the contents transferred to a dish, and the liquid evaporated to dryness at 100° C. It might have been better to neutralize the milk previously to evaporation, but as a matter of fact this had not been done. At the same time that this series of tubes were sealed, two other series were prepared by adding to the same milk one-fourth of its measure of tap-water in the one case, and canal-water in the other. It was thought that these would represent pure and impure waters respectively. The diluted milk was then sealed up in quantities of 10 grammes, as in the case of the pure milk, and one tube of each series opened every week. The experiments, which were conducted at

his request by Mr. Haywood Court, were still in progress, but the following general results might be mentioned. The solids in the case of pure milk fell in fifty-two days from 12.58 to 11.21 per cent., being a loss of 1.37 per cent. The diluted milks lost in the same time 1.44 and 1.46 per cent. respectively. These experiments do not show the varying rate of change which has been observed in other cases to be produced by dilution or the addition of impure water. On the other hand, they exhibit a marked disagreement with the change alleged by the Somerset House chemists to occur in milk by keeping. Had their notorious time allowance for change been correct, the loss of solids in fifty-two days would have been 0.68 per cent., while the amount actually lost was exactly twice as much.

The variable rate of change in milk by keeping was discussed in an able paper read before the Society some years since by Mr. Stokes (*ANALYST*, xii., 226), and in the discussion numerous figures were given conclusively proving the utter worthlessness of the referees' time allowance for change. The Somerset House system of constant allowance was perhaps the most preposterous practice ever devised by persons occupying a responsible position, and it was pitiable to reflect that it had been adhered to for a long series of years, in spite of its palpable absurdity and its repeated impeachment by all those best qualified to judge. The referees' time allowance might well be permitted to sink into the obscurity it deserved, were it not that Mr. Bannister, in his evidence before the Committee on Food Products Adulteration, said that it gave substantially the same results as the system of compensation now employed, which latter he insisted was thoroughly trustworthy. He admitted that the presence of a preservative affected the rate of change, but said that the two processes gave substantially the same results. This would appear to mean that the present plan gave results agreeing with those of the time allowance, no matter whether a preservative had been added or not. This was tantamount to the proposition that things which are equal to the same thing are not equal to each other. As to the revised version of allowance-making, he would merely point out that its inventors had not ventured to present it for discussion before any competent tribunal or to publish the details in scientific journals; and as they were very easily satisfied in the case of the time allowance, it was probable that they were content with a minimum of accuracy in the results of their new system.

It must be admitted that the Somerset House Chemists had been placed in a very difficult position by having submitted to them samples of milk which every chemist knew were unfit for analysis. If, instead of pretending to accomplish impossibilities, the referees had eighteen years ago certified in intelligible terms their inability to form a definite opinion on the samples of milk submitted to them, the law would very quickly have been modified. In only four cases, he believed, in the course of eighteen years had the referees certified that samples of milk submitted to them were not in a fit condition for analysis, and these cases were comparatively recent.

The evidence given by Mr. Bannister was a revelation to public analysts in many respects, but not even his ignorance of the Society's limit for fat astonished them more than his statement that the words "we are unable to affirm," which have caused so much heart-burning, were intended by the referees to indicate to

the court that they could not express a definite opinion on the sample either one way or the other! It would be interesting to ascertain how many scores of failures of justice were directly attributable to the misleading or ambiguous wording of the referees' certificates, and how many real cases of non-confirmation of public analysts' certificates would remain after deducting the former class.

With regard to the preservation of milk samples, Mr. Allen said that a portion of every adulterated sample which passed through his hands was preserved by adding to it twice its weight of alcohol. In one case, the sample so preserved was analysed after several weeks by Mr. Hehner and Mr. Estcourt, and their results served to refute the certificate given at Somerset House, and prevent the failure of justice which would have resulted had the referees' certificate been accepted. But he was not satisfied with the use of alcohol as a preservative, and had tried several other substances, with very limited success. Bichromate of potassium had been advocated, and had been thoroughly tried in his laboratory by Mr. W. G. Wagner, but the very considerable quantity of it required made it objectionable; otherwise it had the advantage that the actual amount which had been used could be ascertained with precision, and due allowance made accordingly. Hydrofluoric acid, chloroform, and carbon disulphide had also been tried, with limited success. What was wanted, in his opinion, was a solid preserving agent which could be added to the milk at the time of purchase in the form of pellets of definite weight, such an addition being legalized. If this were done in every case, the referees would receive their portions of the sample in a practically unchanged condition, the chief bone of contention with Public Analysts would be buried, and the grave loss of caste and credit which Somerset House had suffered through its ill-advised action in the past might ultimately be forgotten.

Mr. Bodmer said that Mr. Stokes had made some experiments which showed the utter unreliability of any such thing as a time allowance, and this confirmed Mr. Bevan's statements. Formaldehyde was now being much used for preserving milk for analytical purposes, under the name of "Formalin."

Mr. Richmond said that he had been using formaldehyde for the last year and a half; 0.05 per cent. would keep milk for a month, and larger quantities almost indefinitely.

Dr. Adams said that he had also used formaldehyde, and had found that the addition of five or six drops was sufficient to preserve a sample of milk for a week. Some of the points referred to in Mr. Bevan's paper had been alluded to in his (the speaker's) paper on the use of ammonia in the analysis of sour milk. He (Dr. Adams) had come to the conclusion that the loss experienced was in some measure due to loss of water of crystallization. We might well believe that the milk sugar might be caramellized by the acids formed during the decomposition of milk, when these acids were concentrated during the drying of the solids.

Mr. Cassal said that he could not agree with the suggestions of Mr. Allen and Mr. Bodmer that an antiseptic should be added to the milk by the inspector when taking the sample. He entirely disapproved of inspectors having anything to do with samples beyond taking them. Any other course would open the door to errors

of all kinds. The addition of a preservative by an inspector would render it impossible to take action successfully against a vendor for using preservatives. The sole object of the suggestion to add a preservative was to enable the Somerset House chemists to make analyses which would enable them to give an opinion approaching to accuracy with respect to the composition of a milk when fresh. The Somerset House analysts should have plainly stated at the outset that it was an absolute impossibility to arrive at a knowledge of the composition of a milk when fresh from an analysis made of the milk when decomposed. After the position they had taken up, and actually defended, it was hardly to be expected that they would now publicly acknowledge themselves to be absolutely wrong by accepting such a proposition. That kind of thing was hardly characteristic of Government Departments. He (Mr. Cassal) had noticed similar, and, in fact, considerably greater, diminution in total solids on keeping milk; there was no regularity about it, and it was unscientific and absurd to suppose that there would be. The differences in the extent of the loss must depend upon the temperature at which the sample was kept, and on the nature of the organisms which had obtained access to it.

Mr. Northfield Yarrow said that he had also noticed a great loss in milk solids when they were not evaporated as rapidly as possible, and he found this out in much the same way as Mr. Bevan had done. Milks were sometimes received late in the day, and the total solids were then weighed out and put inside the oven of the water-bath at once. The total solids thus obtained were much below those of the same milks which were evaporated outside the bath immediately after weighing. Sometimes the loss was as much as 2 per cent. With regard to formaldehyde, the 40 per cent. solution had been in use in Mr. Stokes' laboratory for over a year for preserving reference samples, and had given great satisfaction. One sample, which nine months ago had had this preservative added, was even then in an apparently fresh condition, but it had lost 0.4 per cent. in the total solids and 0.2 per cent. in the fat. For various reasons very little reliance was placed on these figures, but some series of experiments were now being made which might do something to show the rate of decomposition in milks when this compound was added.

Mr. Allen said that they must always have referees, and although they might not always see eye to eye with the gentlemen who at present occupied the position, they ought certainly to give them the utmost assistance in their power. It would be impossible for them to get their samples of milk in an undecomposed state until the addition of a preservative at the time of purchase was legalized. The nature and method of addition need not be specified by Act of Parliament, but might well be left to the Board of Reference, the formation of which they advocated, and then duly confirmed by Order in Council.

Mr. Hehner said he quite agreed with Mr. Cassal that inspectors should not be allowed to tamper in any way with samples. It was essential that an unaltered sample should be handed to the referees. But he did not think that the law laid on the referees the duty of giving an opinion on decomposed samples. He knew, indeed, that the referees had occasionally declined to examine samples which in their opinion were unfit for analysis; so, clearly, they were not under legal com-

pulsion, and he thought that it was never intended that such should be the case. The analysis of decomposed milk-samples was a proceeding which the Somerset House chemists had taken entirely on themselves; and it was a pity they did not at the beginning refuse to analyse decomposed samples, or, having analysed them, had not clearly stated in their certificates that they could not come to any definite conclusion. Public Analysts would certainly never have ventured to have given opinions on samples five or six weeks old—a course which had been regularly pursued at Somerset House. If the referees had been what they ought to be, truly scientific advisers of the Justices, they would have made some suggestion similar to Mr. Allen's. The system of allowances had been nothing short of scandalous. The Society had discussed them on many occasions, and Mr. Bevan had helped to clear up the subject. Officials, perhaps, could not be expected, after persisting in making erroneous allowances for seventeen or eighteen years, to confess now that they had been wrong all the time. We did not know with absolute certainty what takes place in the best-known fermentations—such as, for example, the alcoholic fermentation. Given a certain quantity of sugar, we did not know exactly how much alcohol and how much carbon dioxide were produced under all possible circumstances. If even this well-known process could not be expressed by a simple formula, what could be said about such products as those of the decomposition of a complex heterogeneous mass of albuminoids and other bodies acted on by an unknown variety of possibly unknown organisms? It was all very well to say that a molecule of milk-sugar was simply converted into two molecules of lactic acid, but no organism works for nothing. In the decomposition which it effects it evidently derives something for its own benefit, and life cannot be carried on without destructive metabolism. There must always be a loss between the stage of sugar and that of lactic acid, and what this is was unknown. Surely the day had gone past for regarding fermentation reactions as having the simplicity of text-book equations. As to the quantity of alcohol produced, we could only approximately calculate the quantity produced back to sugar; and he would like to know what the acetic acid was to be calculated into. Where did the acetic acid come from? From the alcohol or from the albuminoids?—the one was a small molecule, the other a large one. If the acetic acid and the ammonia were to be calculated into albuminoids, we got back to the latter from two sources. It was utterly unjustifiable to pretend to be able to make corrections for decomposition, and to compare the, at best, approximate figures thus obtained with those derived from the analysis of samples when fresh. From a merely analytical point, he would like to know how small quantities of acetic acid could be accurately determined in such complex bodies as the products of decomposed milk. The suggestion made by Mr. Bannister, in his evidence before the Parliamentary Committee, that such corrections for decomposition did not in any way concern the Public Analyst, illustrated his inability to appreciate the difficult position of the Public Analyst, who clearly was deeply concerned in a proceeding which affected his reputation.

Mr. Bevan, in reply, said that he agreed that the suggestion of Mr. Allen as to the inspectors being empowered to add preservatives was one to be deprecated.

Some arrangement by which the sample should be at once submitted to the referees would appear, however, to be desirable.

Several members here pointed out that such a course would involve a large expenditure, to which Mr. Bevan replied that possibly, then, as there were relatively few adulterated milks referred to Somerset House, whenever samples were found by the Public Analyst to be adulterated, the duplicates might be at once sent to the referees, if so desired by the vendor, without waiting for decomposition to progress.

In conclusion, Mr. Bevan pointed out that in his experience, as well as in that of others, the present referees under-estimated the fat in milk, and over-estimated, to an even greater extent, the solids-not-fat.

Estimation of Fat in Cheese. Stefan Bondzynski. (*Zeit. für Analyt. Chemie*, 1894; *Zweites Heft*, pp. 186-189.)—This is the application to cheese analysis of the Werner-Schmidt method of estimating fat in milk. A weighed quantity of the finely-divided cheese is placed in the tube, and decomposed with 20 c.c. HCl of sp. gr. 1.1, containing about 19 per cent. HCl. On cautiously warming over wire gauze the melted fat rises to the surface. After cooling, 30 c.c. of ether are added, and the tube warmed very gently, until the HCl and ethereal solution of fat separate sharply. Centrifugal force helps this, but is not essential. After the volume of ether has been read off, 20 c.c. are pipetted into a weighed Erlenmeyer flask. From this the quantity of fat in the entire solution can be calculated. The advantages claimed for the method are that it is rapid, and easily carried out.

The following results of cheese analyses are given :

	I.	II.	III.	IV.	V.	VI.
(1)	31.52	32.37	32.58	2.53	2.60	0.96 per cent. fat.
(2)	31.54	32.45	32.61	2.55	2.65	0.98 " "

The fat in Cheeses I. and III., estimated by extraction in a Soxhlet, gave 31.43 per cent. and 32.62 per cent. respectively. Cheeses IV. and V. were made from centrifugal-skimmed milk. Cheese VI. had had its fat partially removed for analytical purposes.

C. A. M.

Estimation of Essential Oils, especially in Cloves and Mace. W. Lenz. (*Zeit. für Analyt. Chemie*, 1894; *Zweites Heft*, pp. 193-200.)—The solubility of oil of cloves in a 50 per cent. solution of sodium salicylate suggested to the author an improvement on the processes used to estimate the essential oils in spices. From 10 to 20 grammes of the substance is introduced into a retort holding 200 c.c., and having its beak inclined upwards, and then bent downwards at right angles at the middle, and connected with a condenser. Sufficient water is added to form a thin paste, and steam is passed through until the liquid distilling is free from every trace of oil. In order to prevent frothing, he introduces 10 c.c. of pure olive oil into the retort before distillation. The distillate (about 500 c.c.) is nearly saturated with sodium chloride, and shaken with successive portions of ether in a separating funnel.

It is usually sufficient to do this three times. The ethereal solution, freed from water by digesting for three or four days with calcium chloride, is evaporated in a weighed flask at 30°, a current of dried air being meanwhile passed through. It is weighed at intervals of five minutes until the weight becomes constant, and the quantity of eugenol is then estimated by Thom's method.*

By substituting a 50 per cent. solution of sodium salicylate for water in the retort, the author found that considerably higher results could be obtained. In the case of cloves, an average of 19.45 per cent. of oil, containing 84.52 per cent. of eugenol, was obtained, as compared with 17.75 per cent. of oil, containing 79.44 per cent. of eugenol, in the water distillation.

With mace, the oil of which is nearly insoluble in sodium salicylate solution, the method yielded an average of 8.41 per cent. of oil, while distillation with water alone only yielded 6.73 per cent. That the increased quantity of oil is not due to the boiling-point of the water being raised above 100° by the sodium salicylate is proved by experiments in which calcium chloride and potassium acetate are substituted for sodium salicylate. In these cases the quantity of oil yielded is almost identical with that yielded by the distillation with water alone. The author suggests as the explanation the peculiar solvent action of the sodium salicylate on the plant tissues which enclose the essential oil.

C. A. M.

The Decomposition of Albuminoids by Alkaline Hydrates. Dr. Victor Vedrödi. (*Zeit. für Analyt. Chemie*, 1894; *Drittes Heft*, pp. 338-340.)—The author's experiments prove—

- (1) That albuminoid substances are decomposed by boiling with sodium hydrate, ammonia being produced.
- (2) That the decomposition is slow, and even after twelve hours' boiling is not complete.

This raises the question: To what extent in tobacco analysis does the quantity of ammonia formed under such circumstances affect the estimation of the nicotine?

C. A. M.

On Lard. Dr. Samuelson. (*Zeit. für Analyt. Chemie*, 1894; *Heft 2*, pp. 189-192.)—Experiments on qualitative tests for detecting adulteration in lard are described at length. The conclusion arrived at is that, since all the recommended qualitative reactions may give negative reactions with a lard known to be adulterated with vegetable oil, the iodine number remains, as heretofore, the only reliable test.

C. A. M.

Method for the Determination of the Freezing-Point of Fatty Acids. F. Wolfbauer. (Translated and abridged by S. S. Emery.) (*Journal Amer. Chem. Soc.*, 1894, xvi, No. 10, pp. 665-670).—To prepare the fatty acids, 120 grammes of the fat are mixed with 45 c.c. of a solution of KOH (1,250 grammes per litre) and

* *Zeit. für Analyt. Chemie*, 90-738.

stirred at a temperature but slightly above its melting-point until emulsified. The beaker is then covered and kept at 100° until the fat is completely saponified. The soap is decomposed with 165 c.c. of dilute H_2SO_4 (specific gravity 1.143), preferably in a silver dish, and the fatty acids are washed, firstly with dilute H_2SO_4 (5 per cent.), and afterwards with water till the washings no longer taste acid. They are then dried in an open dish for two hours at 100°.

In the determination of the freezing-point, a thin-walled test-tube, 3.5 c.m. by 15 c.m. is fitted by means of a cork into a suitable bottle. A centigrade thermometer, graduated in fifths of a degree from 1° to 60°, is fixed into this tube by a cork loose enough to allow the contents of the tube to be stirred. The tube is filled with the melted acids to within about 1 c.m. from the top, and these are slowly stirred until partial solidification sets in, when the thermometer is rapidly turned round ten times and then allowed to stand, care being taken that it clears the bottom of the tube by 4 or 5 c.m. The highest temperature to which the mercury rises is taken as the freezing-point. Duplicate determinations should not differ more than 0.1° C.

Experiments which led to the adoption of this method showed that the method of saponifying did not affect the melting-point, but that when alcoholic saponification was used, boiling the soap solution one and a half hours was necessary to completely remove the alcohol. The length of time in saponifying did not influence the final result.

It is essential that the fatty acids should be well dried, water lowering the freezing-point and concordant results not being obtained. Two determinations, using the same undried acids, gave melting-points of 43.14° and 42.86°. It is immaterial whether the dried acid be used immediately for the determination or be allowed to solidify and subsequently remelted.

The following determinations of the melting-point of the same fatty acids, using tubes of different sizes, prove that the test-tube may exceed 3.5 c.m. in diameter, but must not be less, otherwise the ratio between heat radiating from the fatty acid through the walls of the tube and the amount of liberated latent heat is disturbed :

			Test-tube 3.5 c.m. diameter.				Test-tube 2.5 c.m. diameter.				Difference.
Fatty Acid 1.	...		43.52°	...			43.34°	...			0.18
„ 2.	...		42.88°	...			42.65°	...			0.23
			Test-tube 3.5 c.m. diameter.				Test-tube 7 c.m. diameter.				Difference.
Fatty Acids	...		43.45°	...			43.46°	...			0.01

The error due to incomplete immersion of the thermometer may be almost eliminated by using a thermometer shortened by having an enlargement blown in the bore in the interval between 2° and 28°, thus diminishing the amount of mercury above the surface of the fatty acid.

C. A. M.

Determination of Volatile and Insoluble Fatty Acids in Butter Fat. W. H. Beal. (*Journal Amer. Chem. Soc.*, 1894, xvi., No. 10, pp. 673-676).—The process consists in decomposing the saponified fat with a 20 per cent. solution of ortho-

phosphoric acid, and expelling the volatile acids by means of a current of steam, the operation being usually complete when 500 c.c. have distilled over. The insoluble acids are washed until free from phosphoric acid, and dried on water-bath at 100° until they begin to increase in weight. Very uniform and concordant results are said to be obtained. The author disclaims previous knowledge of Goldman's method of distilling the volatile acids in a current of steam.*

C. A. M.

The Influence of Alum, Aluminium Hydroxide, and Aluminium Phosphate on the Digestibility of Bread. W. D. Bigelow and C. C. Hamilton. (*Journal Amer. Chem. Soc.*, xvi., No. 9, pp. 587-597.)—The authors have studied the question with especial reference to the double digestion of the gastric and pancreatic ferments. Their general method is the determination by Kjeldahl's process of the quantity of albuminoids in the material left undissolved after treatment with artificial pepsin and pancreas solutions.

Five processes are used :

1. *Digestion in Pepsin Solution.*—1 gramme Merck's granulated pepsin dissolved in 1,000 c.c. of 0.33 per cent. HCl, and 2 grammes of dried bread previously extracted with ether placed in flask with 100 c.c. of the acid pepsin solution. This is kept at 40° on water-bath for twelve hours, with frequent shaking. The contents are then filtered, and the residue washed, dried, and Kjeldahled.

2. *Stutzer's Method.*—2 grammes of bread are digested in the pepsin solution, and afterwards for six hours in Stutzer's pancreas solution.

3. *Stutzer's Method modified by Wilson.*—After treatment with the pepsin solution, the residue is digested for twelve hours at 40° in pancreas solution, made by dissolving 1.5 grammes Merck's pancreatin and 3 grammes sodium carbonate in 1 litre of water.

4. *Niebling's Method.*—2 grammes of the bread washed with ether and placed in flask with 100 c.c. of 2/10 per cent. HCl. Boiled for fifteen minutes, cooled, neutralized, or made slightly alkaline with sodium carbonate solution. 100 c.c. of Stutzer's pancreas solution then added, and the flask immersed for six hours in water-bath at 37° to 40°.

5. *Niebling's Method modified.*—The same as the preceding, except that the pancreatin solution given under Wilson's modification of Stutzer's process is used instead of Stutzer's pancreas solution.

The results of digesting bread free from alum are compared with those from bread containing known quantities of alum, aluminium hydroxide, and aluminium phosphate :

I. *Bread Free from Alum.*—Loaves made from flour known to be pure were cut in slices, dried at 98°, ground and bottled. They contained 12.06 per cent. albuminoids. The following percentages of the albuminoids were found to have been digested by the different methods :

Pepsin Solution.	Stutzer's Method.	Stutzer's Method modified by Wilson.	Niebling's Method.	Niebling's Method modified.
93.26	93.57	93.21	93.38	93.28

* *Analyst*, September, 1892, 174.

II. *Alumed Bread*.—Two loaves made from alumed flour. No. 1 contained 0·8 grammes of alum and 11·88 per cent. of albuminoids. No. 2 contained 4·28 grammes of crystallized alum and 12·06 per cent. albuminoids. Percentages of albuminoids digested :

	Pepsin Solution.		Stutzer's Method.		Stutzer's Modified.		Niebling's Method.		Niebling's Modified.
1.	89·11	...	92·56	...	92·21	...	92·54	...	92·74
2.	80·98	...	92·4	...	92·44	...	—	...	92·62

From these results the authors conclude that the influence of alumed flour on the digestion is over-estimated, since the albuminoids not digested by the pepsin are almost all digested by the alkaline pancreas solution.

III. *Bread containing Aluminium Hydroxide*.—Loaf No. 1 contained ·54 gramme ; No. 2, 2·5 grammes. Percentages of albuminoids digested :

	Pepsin.		Stutzer's Method.		Stutzer's Modified.		Niebling's Method.		Niebling's Modified.
1.	87·03	...	92·18	...	92	...	91·77	...	91·90
2.	86·78	...	90·43	...	90·21	...	89·13	...	88·96

IV. *Bread containing Aluminium Phosphate*.—Loaf No. 1 contained ·64 grammes ; No. 2 contained 3·2 grammes. Percentages of albuminoids digested :

	Pepsin.		Stutzer's Method.		Stutzer's Modified.		Niebling's Method.		Niebling's Modified.
1.	80·87	...	83·11	...	82·56	...	86·35	...	86·46
2.	71·21	...	78·26	...	81·32	...	82·18	...	81·74

The influence of aluminium hydroxide on digestion is thus shown to be about the same as that of an equivalent quantity of alum. The action of the phosphate is quite different, for, in spite of frequent statements as to its insolubility, the preceding results show that from 10 to 12 per cent. of albuminoids which are digestible in presence of aluminium hydroxide and alum are insoluble in the presence of an equivalent quantity of aluminium phosphate.

C. A. M.

Separation of Tin and Antimony in Alloys. Mengin. (*Comp. Rend.*, 1894, cxix. 224 ; through *Chem. Zeit.*).—Tin and antimony are first separated from the other constituents of the alloy by treatment with nitric acid. The mixed oxides are weighed and treated with hydrochloric acid and a rod of tin. On heating, the antimony is precipitated as metal, and the tin goes into solution as stannous chloride. The metallic antimony is washed with boiling water, dried, and weighed, the tin being obtained by deducting this value after calculation into oxide from the weight of the mixed oxides of tin and antimony originally obtained.

B. B.

NOTE BY ABTRACTOR.—The method in its present form is vitiated by the facts (1) that antimony is not completely separated as an insoluble oxide on treating an alloy containing it with nitric acid ; and (2) that metals other than antimony and tin are invariably retained by the oxides left on dissolving an alloy of this class in nitric acid. Accurate separation of the antimony and tin from the remaining metals of the alloy is, therefore, requisite before using the reduction process described above.

B. B.

Distinctions between Atropine and Strychnine by means of Vitali's Reaction. D. Vitali. (*Boll. Chim. farmac.*, 1894, xxiii. 449; through *Chem. Zeit.*).—When strychnine is oxidized with nitric acid, and the dry residue treated with alcoholic potash, it gives a red colour, which may possibly be confused with the similar reaction for atropine, which has been described by the author. The following distinctions exist, however: (1) On oxidation with nitric acid, especially immediately after the evaporation of the acid, atropine gives an agreeable odour, while strychnine under the same circumstances yields no smell. (2) Strychnine becomes yellow on oxidation, and the residue left on evaporation is also yellow; no such coloration occurs with atropine. (3) On evaporating the alcohol, after the addition of alcoholic potash, atropine gives a violet residue, which becomes more intense in colour on again treating it with alcoholic potash; with strychnine the residue is yellow or reddish-yellow, and becomes reddish-violet on repeated treatment with alcoholic potash. (4) When water is added after the colour has been produced by alcoholic potash, the coloration due to atropine is bleached, but that given by strychnine becomes yellow. (5) When atropine is oxidized with nitric acid, and the dry residue treated with ammonia, yellow drops are formed which become violet on the addition of alcoholic potash. Under like conditions strychnine gives with ammonia an orange-red, and when alcoholic potash is added, the violet colour produced is only transitory, becoming a strong blood-red. On shaking out the ammoniacal solution of the oxidation product with chloroform, and evaporating the solution in chloroform thus obtained, the residue given by atropine is nearly colourless, and takes a permanent violet colour when treated with alcoholic potash. Strychnine, on the other hand, when submitted to the same process, colours the chloroform slightly yellow, and yields on evaporation a yellow residue, which gives a strong orange-red coloration on addition of alcoholic potash.

B. B.

Contributions to Volumetric Analysis. B. Reinitzer. (*Zeitschrift für angewandte Chemie*, 1894, pp. 547-551, and pp. 573-579.)

Indicators.

In the opinion of the author litmus solution is the most serviceable indicator, excelling methyl orange in sharpness of change of colour and sensitiveness (about eight times as great), while it possesses an advantage over phenol-phthalein in being capable of being used in the presence of ammonium salts.

The disadvantage in its use is its great sensitiveness to carbonic acid, and special precautions are accordingly necessary in its preparation and use to avoid this source of error. It should be prepared from good litmus, poorer samples in the market being often almost useless, and since this contains alkaline carbonate, the solution must be boiled for seven or eight minutes and then neutralized with HCl, so that the wine-red colour remains even on further boiling. The solution is then cooled and an equal volume of strong alcohol added. The stock solution should be kept in a bottle with a delivery pipette inserted through the cork.

The final change of colour is sharpest when the liquid to be titrated is boiled for seven or eight minutes and then well cooled. In order to avoid the influence of atmospheric CO_2 it should not be allowed to stand exposed for long, and dilution with unboiled distilled water should be avoided. A similar source of error is that carbonate often occurs in standard alkaline solutions. The author found the same in calcium and barium hydrate solutions. In one litre of clear lime water, containing 1.1738 gramme of CaO , there was found .0093 and .0087 gramme of CO_2 . To avoid this it is necessary to so arrange that after boiling only a little alkali need be added.

Experimental proof of the inferiority of methyl orange as an indicator is given, showing that, owing to the fact that the final change of colour is not sharp but passes through intermediate changes, it cannot be used for accurate work with decinormal solutions, nor when the liquid to be titrated is of large volume (500-1,000 c.c.). In the former case, in titrating 250 c.c., twenty to thirty drops of decinormal acid are required to change the colour from clear yellow to the clear "onion" red; in the latter, even with only 100 c.c., six drops of decinormal acid = 0.19 c.c., are required for the change. On the other hand one drop decinormal alkali or acid is sufficient to change the colour of 250 c.c. of a neutral solution when litmus is the indicator.

Emphasis is laid on the point that the fluid must be cold when titrated, whatever indicator be used. Thus, a solution, which when cold only requires two drops = 0.06 c.c. of decinormal acid to change the colour of the litmus, requires five drops = 0.15 c.c. when heated to 100° . Similarly, phenol-phthalein is three times more sensitive in a cold solution than in a hot one. In the case of methyl orange a solution of 250 c.c., which shows a change of colour with sixteen drops of decinormal HCl = .48 c.c. in the cold, requires 2 c.c. of the acid when heated.

It is suggested, as a possible explanation of this, that with the rise of temperature an increasing dissociation of the combination between the indicator and the acid or alkali occurs, so that the excess of either necessary to produce the colour also rises with the temperature.

Preparation and Use of Pure Na_2CO_3 as a Standard for Acids and Alkalies.

Pure Na_2CO_3 may be obtained by the following method: As much sodium bicarbonate as possible is dissolved in 250 c.c. of water at 80°C . and freed from insoluble impurities by filtration. On cooling down to a temperature of from 10° to 15° in a stream of water a double salt which has the composition of $\text{Na}_2\text{CO}_3 + \text{NaHCO}_3 + 2\text{H}_2\text{O}$ crystallizes out. Most of the soluble impurities remain in the mother liquor, which is sucked off, and the salt is further purified by repeated washing with small quantities of cold water. When heated in a platinum basin at a temperature just below perceptible redness, the residue is perfectly pure Na_2CO_3 .

The salt thus prepared should always be heated again immediately before use and the weighed quantity dissolved in warm water. In accurate determinations the temperature of the solutions must be taken into account: a liquid which measures 50 c.c. at 17°C . becomes 50.05 c.c. at 22° , which causes an error of 0.1 per cent.

Ammonium Chloride as a Standard for Acids, Alkalies, and Chloride Solution.

The salt is easily obtained pure, either by careful selection from the commercial salt, or by neutralizing ammonia solution with HCl. Its purity is tested in solution by ammonium sulphide, and by sublimation. When using it for standardizing acids the author employs a modification of the ordinary method of estimating ammonium in its salts. About 2.6 grammes are placed in the distilling flask, which is then about half filled with water. A stick of potash weighing about 10 grammes is introduced, and the flask heated with a Bunsen. The NH_3 liberated is conducted by a tube delivering downwards into a flask containing the acid to be standardized. About fifteen minutes is sufficient for all the gas to pass over. The receiving flask is boiled for six or eight minutes to get rid of CO_2 , cooled, and the unneutralized acid titrated back with a standard alkali.

As one example of the accuracy of the method the following may be quoted: 2.68692 grammes NH_4Cl by calculation correspond to 50.336 c.c. normal acid, while the amounts experimentally found were 49.71 and 49.74 c.c.

Too large an excess of potash in the distilling flask is to be avoided, since it increases the difficulty of expelling the ammonia.

In standardizing alkalies with ammonium chloride, the salt is placed in a flask of Schott's glass, 300 c.c. of water added, and an excess of the alkali. The flask is boiled until the escaping steam no longer smells of ammonia (fifteen to twenty minutes). Acid is run in to acid reaction and the flask boiled, cooled, and titrated back. The results tally with those obtained by the distillation method described above.

Influence of Boiling on Glass Vessels.

That a boiling fluid dissolves alkali in glass is shown by the following experiment:

To 300 c.c. of distilled water, placed in an ordinary boiling-flask, lime-water was added, and after boiling and cooling titrated with decinormal HCl. On boiling for fifteen minutes .03 c.c. additional acid was required, and the same quantity on again boiling for fifteen minutes.

With larger quantities of liquid the error caused by boiling for fifteen minutes was so increased that the advantage of using a decinormal acid was quite lost. 700 c.c. of distilled water boiled for four hours in a new Erlenmeyer flask required 9.53 c.c. of $\frac{1}{10}$ N acid to neutralize the dissolved alkali. After continuing the boiling for fifteen minutes the reading was 10.22 c.c., rising to 10.92 c.c. when again boiled for the same length of time.

A Bohemian hard-melting potash glass offered about ten times the resistance of ordinary glass. After four hours' boiling 700 c.c. required 1.09 c.c. $\frac{1}{10}$ N acid to neutralize the dissolved alkali, rising after ten minutes' longer boiling to 1.14 c.c., and to 1.16 c.c. after a subsequent seven minutes. Thus the amount of alkali dissolved by boiling liquids for not more than ten minutes in vessels of this kind of glass affects the accuracy of the result but slightly.

It was not, however, until he used vessels made of Dr. Schott's new Jena glass that the author was able to obtain perfectly satisfactory results in standardizing

solutions. In a flask of this glass 700 c.c. of distilled water boiled for four hours used 0.13 c.c. $\frac{1}{10}$ N acid, which after eighteen minutes' further boiling became 0.18 c.c.

C. A. M.

The Detection of Chlorine in the Presence of Bromine and Iodine.
A. Nieliers and M. Fayolle. (*Comptes Rendus*, 1894, cxviii., 1152 and 1204, through *Chem. Zeit.*)—The authors have devised a method for the detection of traces of chlorine, in presence of much bromine and iodine, which depends upon the relations of the three halogens to an acid solution of aniline. In such a solution iodine produces no apparent change, bromine causes the formation of a white precipitate, while chlorine gives a colored product varying according to its quantity from a definite black precipitate to a blue coloration. The delicacy of the reaction depends on the acidity of the aniline solution. The best results are obtained by the use of a mixture of 400 c.c. of a saturated aqueous solution of aniline and 100 c.c. of glacial acetic acid. This mixture can be kept in yellow bottles, and then remains free from colour. The process of analysis is as follows: 10 c.c. of the solution to be examined are placed in a flask, and 5 c.c. of a mixture of equal parts of sulphuric acid and water, and 10 c.c. of a saturated solution of potassium permanganate are added. The flask is gently warmed, and the vapour therefrom caught in a well-cooled receiver containing some of the aniline solution specified above. If chlorine alone be present in the original solution, as little as 0.1 milligramme gives a perceptible blue coloration, which on warming quickly becomes red. Iodine is oxidized in the flask and does not distil. Bromine, however, comes over and forms a precipitate in the aniline solution, the precipitate being colored only if chlorine be also present. The presence of bromine interferes considerably with the delicacy of the reaction from the formation of bromine chloride. This drawback is avoided by the following preliminary treatment: The halogens in the solution to be tested are first precipitated in the ordinary way by silver nitrate, and the mixed halogen salts of silver thus obtained are washed and allowed to stand for some time under weak ammonia solution (1 c.c. of strong ammonia diluted with 10 c.c. of water). The silver chloride is dissolved, the bromide and iodide being but little affected. The solution is filtered, the ammonia evaporated, and the silver thrown down with sulphuretted hydrogen, and the solution filtered. The excess of sulphuretted hydrogen is then driven off by heating, and the solution concentrated to 10 c.c., and put through the process already described. It should be noted that a cork should not be used for the distilling-flask, but a ground-in stopper carrying a delivery-tube and a safety-funnel. By this modified method the smallest trace of chlorine can be detected in bromine and iodine compounds. In the case of the presence of cyanogen this substance is removed before the examination for halogens is begun. B. B.

EXTRACTS FROM THE EVIDENCE GIVEN BEFORE THE SELECT
COMMITTEE ON FOOD PRODUCTS ADULTERATION, ON
JULY 11, 18 AND 25.

MR. RICHARD BANNISTER.

(Continued from page 231.)

5. EVIDENCE REFERRING TO MARGARINE IN BUTTER.

607. With regard to butter, you have changes taking place in butter ordinarily when it is kept for any length of time, have you not, which makes some difficulty in analysis?—There is a very great difficulty in the analysis of butter.

608. In what way?—Because, keeping the butter, it gets rancid, and we find that the insoluble fatty acids increase rather largely and the soluble fatty acids diminish; and inasmuch as if the insoluble fatty acids are high, the probability is that foreign fats may have been added, it follows as a matter of course that if you get a fresh butter to which fatty acids within certain limits have been added, the result of the analysis may be the same as the examination of an old butter that is perfectly genuine.

609. That introduces confusion and causes a difficulty of detection?—Very great difficulty of detection.

610. Butter was, I think, the next most frequent article submitted to you after milk?—Yes.

611. And the chief difficulty that you find in the butter analysis is the detection of foreign fat?—Just so.

613. In what form is the foreign fat introduced?—It is rather difficult to say, because there are so many ways of introducing this foreign fat, but very often it is introduced in the form of *stearine* or *margarine*.

627. Where do the butters come from chiefly that are adulterated with foreign fats, such as *margarine*?—Some from France and some largely from Holland.

628. And from Belgium?—Chiefly from Holland.

666. *Margarine*, I think, in its purest form is practically refined beef-fat, is it not?—It is.

667. Is it, then, fat of the same nature which exists in milk?—Yes.

685. But in inferior kinds of *margarine* coarser oils are used, such as those of the coco-cotton plant and the palm, which are substituted for the finer oils?—And a large quantity of sesame oil is used too.

686. And these are mixtures that are sold in the market sometimes as *butterine*-*porcine*; have you heard of that, or *lactine*, or *cocoa-butter*, or *lardine*; have you heard these expressions used?—Yes, I have heard those expressions.

687. Those represent the inferior kinds of *margarine*?—Yes.

688. And those are either mixed with butter or churned with milk, and they make up some of the various compounds sold as *margarine*, as we have heard?—Yes.

1779. The Minister of Agriculture asked you the other day whether you had ever heard of an article called *lactine*, or *cocoa-butter*, or *lardine*, and as to whether these articles were inferior kinds of *margarine*. Do you consider that those articles enter into the composition of *margarine* at all?—*I do not know.*

1780. Is not this lactine refined cocoanut-oil?—It is.

And lardine is a lard substitute?—Yes.

1783. And not in any way whatever sold as a low character of margarine?—*It is not.* (See reply to question 687, page 259.)

959. What is arachis; what substance is it taken from?—*I do not know its origin.*

691. What percentage is the lowest that it is possible to detect of animal fat in butter?—It is a very difficult question to answer, because very frequently there are certain substances used with the animal fat for a particular purpose, and on account of the presence of those substances you may detect a very small percentage, when, if you depended entirely upon the animal fat you could not detect it; you could not say positively that it was there.

692. Will you explain that last answer a little more?—Certain of these vegetable oils that are used in the manufacture of margarine have distinct chemical reactions, and if they are present, of course, the presence of this oil assists you in the determination of the presence of fat.

693. You say that the presence of these oils indicates the presence of fat; but it could not enable you to estimate the percentage of margarine itself, could it?—Very frequently when examining a sample of butter that contains a certain percentage of foreign fat you are quite certain from the analysis that there is something wrong with the butter; that it is either an old butter or something of the kind; and then you may get these indications as you go on in the analysis of samples that determine you, before you come to the end of your examination, that margarine is really present in a sample of butter.

699. Mr. Herbert Gardner asked you about the use of preservatives in milk and butter, and you mentioned especially boracic acid; I suppose that is in the form of the boro-glycerine?—*It is generally used in that form.*

706. I ask you as a matter of fact, would you treat the presence of a little boracic acid, if you found it in butter, differently from the presence of some chloride of sodium?—I should not.

934. I want to ask you a few questions as to the test you apply (I do not think that you have given any evidence on that point) to determine the presence of margarine in alleged butter. Perhaps, as you have given the tests with regard to milk, it would be of value to the Committee if you could give briefly in clear popular terms what tests you consider satisfactory with reference to determining the presence of foreign fats and the proportion of foreign fats in any sample of butter?—Perhaps it would be better to start with the general principle first. In a butter we have there a certain percentage within certain limits of insoluble fatty acids, and a certain proportion within certain limits of soluble fatty acids. In a foreign fat nearly the whole is insoluble fatty acids. It follows that if a foreign fat be added to butter it will increase the percentage of insoluble fatty acids and diminish the quantity of soluble fatty acids. In the analysis of the sample we determine the quantity of the proportion of insoluble fatty acids, and also the proportion of soluble fatty acids. When we have these percentages we have to go back to the variations in genuine butters in the insoluble and soluble fatty acids; then from the physical conditions of the sample,

from the results of the analysis, we have to determine whether the sample that we examine is a genuine butter or whether it contains foreign fat.

935. Can you tell that with absolute accuracy?—You cannot, I am sorry to say, because the butter varies largely in composition itself.

936. I suppose that the fat in milk and the fat in the bullock would be the same?—To a certain extent—not entirely; you get a slightly different composition in butter from what you get in the fat of the bullock.

937. I think in your previous answers you said that margarine exists in milk?—Certainly.

938. The chemical fatty acids exists, do they not, in all these dairy products?—Yes; but when you come to ordinary margarine, in an ordinary butter, there is something else besides that; the fats of margarine do exist in butter, plus the soluble parts that do not exist in the margarine.

939. Do the insoluble fatty acids exist in the milk?—Yes.

941. Plus other substances?—Yes; plus other substances.

944. How do you determine the proportion of foreign fats present?—We can only do that when we get beyond certain limits.

945. What are these limits?—I will give you a case in point: If you take a fresh butter, the probability is that you will get of insoluble fatty acids about 87 or 88 per cent.; in the case of a fat, you will get of insoluble fatty acids something like 96 per cent.; and when you go on the basis of calculation of, we will say, 87 to 96, 9 per cent. represents all the difference that there is between the genuine butter on the one side, and margarine on the other side. Now, in the case of butter that has been kept you may have your insoluble fatty acids go up as high as 89 per cent.; it follows, then, as a matter of course, if you take your first 87 per cent., that you have got 2 per cent. there in the butter of change, and that may be either due to the change of decomposition in the butter itself, or it may be due to the addition of foreign fat; but the 2 divided by 9 shows a 20 per cent. margin for adulteration.

1757. So that it must follow that if you get a fresh butter to which margarine, in certain limits, has been added, the result will be the same as the examination of old butter that is genuine?—Yes.

1759. Of course the analyst would have to state on his certificate whether the butter, as in the case of milk, had undergone any changes that might interfere with the analysis?—Yes.

1760. And he would be in a preferable position for correct analysis, dealing, as he would deal, with perfectly fresh butter?—He would have the butter in a physical condition that would certainly be better than when we got it further on, if it had not been kept properly.

950. Are you satisfied with the test that is called by the other name of saponification?—*That is the test I have been describing.*

951. And which you adopt?—Yes.

952. Does not that work out in figures?—Yes; but only in the limits I tell you, because the butter itself may be so different in constitution owing to its physical condition that it gives so much more margin for the addition of foreign fat.

953. Does the condition make a very considerable difference in the amount of

potassium which is absorbed?—Yes, it does, because that has all to be worked out into percentages again.

954. I see that 2·27 milligrammes is used in America as about the standard of pure butter?—That may be taken as the standard of pure butter, but at the same time they take into account the alterations in the physical condition of the butter, and therefore they cannot make a sharp line.

955. You throw serious doubt on the wisdom of American analysts and other analysts, like Hehner, who have employed these tests, in selecting as the figure 2·27 milligrammes of potassium hydroxide. What is the test that you would apply?—I should like to look into the whole question if you will allow me. If you will ask me the question on Wednesday next I shall be happy to answer it.

(Note the confusion between the different methods of analysis, and the acknowledgment, on the part of the Referee under the Food Act, that he has to look the matter up before being able to reply.)

957. There is one question that I want to ask you with regard to these tests which I think you will be able to answer at once; that is, that those oils, to which Mr. Herbert Gardner drew your attention in his examination, will require a higher percentage of potassium than pure butter, will they not?—Yes.

(Note.—After looking the matter up, Mr. Bannister again refers to the subject as follows:)

1525. There was a question which you wished to consider further with regard to the testing of the fats by the saponification method, whatever that may be; I asked you a question as to the figures used by the American analysts, and especially as to the number of 227 milligrammes of potassium hydroxide to test the quantity that butter would absorb, as compared with other fats that absorb less; and I think you said that if I asked you the question to-day you would answer it?—I shall be very happy to do so. The 227 milligrammes there is mentioned in connection with a test called the Koettstorfer's test, and the number is 227; that refers to butter-fats. But when we come to look at the table itself we find that 227 is the average of the butter experiments; the minimum is 221·5, and the maximum 232·4. I think you will find that the average of those two figures is 226·9.

1526. May I ask you what range these experiments appear to have been taken over, a very large range, or over a limited range?—Over a very large range of different kinds of butter, and also of different kinds of fat. I thought you would like me to work out the result as given above, when I explained the difficulty that there was of determining a small quantity of foreign fat in butter. If we take these figures, the minimum figure of 221·5 and the maximum figure of 232·4, between them you would be able to put into the better description of butter about 30 per cent. of foreign fat to make it come up to the higher one.

1527. That amount could be let in without detection?—Without detection.

1528. Would it be of use if you were to state the average figures for the other fats?—*I can scarcely understand about the other fats; lard oils and the oils to which the test was applied.*

1529. The oil, I understand, takes up a larger quantity, but the organic fats which are used for adulteration take a less quantity, I understand?—Koettstorfer's

number for the butter-fat is 241 to 253, and in the case of arachis, or earthnut-oil, it is 285 to 296; but so far as this test is concerned, after looking it over very carefully, and trying it ourselves, and also after comparing the results of other examinations by other chemists, it is put on one side as not being a sharp test for the detection of foreign fat in butter. (Note the confusion between milligrammes of KHO and the saponification equivalent.)

1534. What is the sharpest test that you do apply?—I think that the sharpest test of all is the estimation of the soluble acids in butter, that is to say, the acids soluble in water.

1535. Is that the test that you refer to when you said that you had to take the physical condition of the butter into consideration, as well as the results of the analysis?—THAT IS SO.

6. EVIDENCE REFERRING TO THE AMOUNT OF WATER IN BUTTER.

615 and 617. During the last two or three years you have had a good many samples (of butter). Have you anything to say about the analysis of these samples?—A system of adulteration that seems to me to have come in lately is putting in too much water; that seems the modern way of adulteration; and there is no doubt that during the last few years there has been a considerable increase in the quantity of water present in butter, in particular kinds of butter.

619. The butters that contain an excess of water are very often Irish butters, are they not?—Yes, they are.

620. And this water has been pressed into them for the purpose of adding to the weight, I suppose?—It has been worked in for the purpose of adding to the weight.

622. What is the standard of water in genuine butter?—There is no standard. In fresh butters it will go from about 12 to 14 per cent. But when we come to salt butter it will go up as high as 16 per cent. or a little more, but those samples contain sometimes as much as 24 per cent.

1770. The strong evidence given before the magistrates at Manchester on the part of Irishmen interested in butter was that it was perfectly consistent with the butter being legitimate that it should contain 20 per cent. of water?—There is no necessity for it to contain 20 per cent. of water.

1776. I have had brought to my notice a certificate given by Dr. Bell in his book, where he included under the head of genuine butter a sample containing as much as 20.75 per cent. of water?—That is a very long time ago, and that was a private sample.

1777. You would not pass a sample now with 20 per cent. of water?—Not without making a remark about it.

1778. You would not pass it as genuine, would you?—No.

2731. Are you aware that Somerset House has passed butter as pure which contained 19 per cent. of water?—No; I do not think we have done so.

2732. Are you aware that Dr. Bell stated that pure butter will sometimes contain over 20 per cent of water?—I think I can explain where we are differing. Those samples which you find in Dr. Bell's book were samples that we had obtained in order to see the quantity of water in commercial butter at that particular time, and

there is no doubt that the sample of butter there mentioned was a pure butter so far as its manufacture was concerned, and that it did contain that quantity of water; but it was not a reference sample.

2733. But it was a pure butter?—Yes.

(*To be continued.*)

REVIEW.

FERTILIZERS AND FEEDING STUFFS. By BERNARD DYER, D.Sc. (Lond.). London: Crosby, Lockwood and Son, 1894. Price 1s.

The little work before us consists of a series of articles from Dr. Dyer's pen which have appeared from time to time in various newspapers, and are now collected and reproduced in book form. To these an appendix is added which contains the text of the Feeding Stuffs Act of 1893 (annotated by A. J. David, B.A., LL.M., of the Inner Temple), and the forms and regulations of the Board of Agriculture. The first chapter is devoted to the consideration of the general functions of fertilizers. The second and third treat respectively on farmyard manure and artificial fertilizers. Chapter iv. deals with the application of artificial fertilizers, and chapter v. treats on purchased feeding stuffs. In chapter vi. the comparative values of feeding stuffs are commented upon. The book, as stated in the preface, is addressed to practical farmers, and not to agricultural students, consequently its contents are couched in plain, homely and familiar language; nevertheless, the information given is solid, sound, and thoroughly reliable. The appearance of the work is extremely opportune, for evidently it is just for want of such information as is here supplied that the Feeding Stuffs Act has been so far practically a dead letter. Any individual of ordinary intelligence cannot fail, after a careful perusal of the work, to be able to form a fairly competent opinion on the value of a fertilizer or feeding stuff from the results indicated by its analysis. The book also abounds in practical hints, not only derived from the subject as considered from a scientific point of view, but also from the author's extensive experience in actual farming operations. It cannot fail to be of the utmost value to those to whom it is specially addressed, whilst the agricultural student, to whom it is not addressed, may peruse it with advantage.

W. J. S.

CORRESPONDENCE.

To the Editors of THE ANALYST.

County Analyst's Office,

Darlington, October 10th, 1894.

SIRS,—The abstract in the current number of *THE ANALYST* which deals with a "Comparison of the Kjeldahl-Wilfarth and Stock Methods of determining Nitrogen," by E. Cavazzani and A. Cecconi, has occasioned me a little surprise. My belief is that these gentlemen have been misled by some error in translation, because in working my method I allow the sulphuric acid to act for some time before adding MnO_2 , and therefore the question of frothing is new to me.

I should not have intruded upon your space with respect to the conclusions at which Messrs. Cavazzani and Cecconi have arrived, if it had not happened that for two years I have used my process exclusively as Analyst to four important Agricultural Societies whose guarantees are of the strictest; and I have never had a single dispute, although my work has been checked by at least half a dozen different analysts acting for the vendors of both fertilizers and feeding stuffs. I venture to think a test like this is a little more to the point than a few experiments made in connection with an evidently imperfect knowledge of the method.—

Yours obediently,

W. J. KEATING STOCK.

THE ANALYST.

DECEMBER, 1894.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on November 7th at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner took the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were duly elected as members: E. A. Hancock, St. Kitts, West Indies; T. A. Pooley, 34, Old Broad Street, E.C.; Dr. J. C. Thresh, Chelmsford; W. G. Wagner, 101, Leadenhall Street; and R. Waterhouse, 101, Leadenhall Street. As associate: Martin Priest, Apothecaries' Hall.

The following gentlemen were proposed for election as members: J. Priestley, B.A., M.D., D.P.H., Leicester; and Raymond Ross, Worcester. As associates: C. A. Mitchell, M.A. (Oxon.), assistant to Mr. Hehner; J. Lewin, assistant to Dr. Bischof.

Dr. Ashby then read the following paper:

ON THE DETECTION OF METHYLATED SPIRITS IN TINCTURES, SPIRITS, OR OTHER COMPOUNDS.

BY ALFRED ASHBY, M.B., F.I.C.

JUST four years ago I read a short paper at a meeting of this Society on "The Detection of Methylated Spirits in Tinctures, Spirits, or other Compounds." It was not published, because I had not then completed my investigation of the subject, and I have deferred making further observations bearing upon it until quite recently.

It is a simple matter which I wish to bring to your notice this evening, but I believe that anything which is calculated to facilitate the work of the public analyst, or to render the detection of sophistications of articles of human consumption more certain or more easy of execution, is fitted for our Society, and will not be without interest to its members.

If, therefore, I can show a more expeditious method for the detection of the fraudulent use of methylated spirits than has hitherto been available, I think you will deem it worthy of your consideration.

The methods heretofore proposed may be divided into two classes: first, those which attempt the detection of methyl alcohol in the presence of ethyl alcohol; and, secondly, those which depend upon the identification of acetone, which appears to be ever present in the wood spirit used for "methylating" alcohol.

It is to the latter class that the method I proposed belongs; but before describing it, perhaps it may be well to recapitulate what has already been done in both those directions.

The following methods are given by Allen in the first volume of his "Commercial Organic Analysis": a work to which one rarely appeals in vain for information on its subject matter.

1. A tedious and complicated process devised by MM. Riche and Bardy for the detection of methyl alcohol in commercial spirit which depends upon the formation of methyl aniline violet. "Ten c.c. of the sample of alcohol, previously rectified if necessary over potassium carbonate, are placed in a small flask with 15 grammes of iodine and 2 grammes of red phosphorus. Methyl and ethyl iodides are formed, and should be distilled off into about 30 c.c. of water. The heavy oily liquid which settles to the bottom is separated from the water, and transferred to a flask containing 5 c.c. of aniline. The flask should be placed in cold water, in case the action should be violent; or, if necessary, the reaction may be stimulated by gently warming the flask. After one hour the product is boiled with water and solution of soda added, when the bases rise to the top as an oily layer, which may be drawn off with a pipette after filling the flask with water up to the neck. One c.c. of the oily liquid thus obtained is next oxidized by adding it to 10 grammes of a mixture of 100 parts of clean sand, 2 of common salt, and 3 of cupric nitrate. After being thoroughly mixed, the whole is introduced into a glass tube and heated to 90° C. for eight or ten hours. The product is exhausted with warm alcohol, the liquid filtered, and made up with alcohol to 100 c.c. If the sample of spirit were pure, the tint of the liquid is red, but in presence of 1 per cent. of methyl alcohol it has a distinct violet shade; with 2½ per cent. the shade is very distinct, and still more so with 5 per cent. To detect more minute quantities of methyl alcohol, 5 c.c. of the coloured liquid are diluted to 100 c.c. with water, and 5 c.c. of this again diluted to 400 c.c. The liquid thus obtained is heated in porcelain, and a fragment of white merino (free from sulphur) immersed in it for half an hour. If the alcohol were pure the wool will remain white; but if methylated, the fibre will become violet, the depth of the tint giving a fair approximate indication of the proportion of methyl alcohol present."

2. The process of J. T. Miller. "In the case of tinctures and other liquids containing fixed matters, the greater part of the spirit should be distilled off and the test applied to the distillate. The method is based on the fact that methyl alcohol produces formic acid when treated with oxidizing agents, but that ethyl alcohol yields a mere trace of the same body. Nevertheless, the fact must not be overlooked that a trace of formic acid (or other reducing agent) is formed, even when pure ethyl alcohol is operated on. Three grammes of bichromate of potassium and 2½ c.c. of concentrated sulphuric acid are mixed in a small tubulated flask with 25 c.c. of water and 3 to 4 c.c. of the spirit to be tested. After standing for a quarter of an hour, the flask is attached to a condenser and the mixture is distilled. When 25 c.c. have passed over, the acid distillate is treated with a very slight excess of sodium carbonate, boiled down to about 10 c.c., and enough acetic acid added to impart a distinct, but feeble, acid reaction. The liquid is then treated with 0.1 gramme of silver nitrate dissolved in about 3 c.c. of water, and the whole gently heated for two or three minutes. If the solution merely darkens a little, but continues quite transparent, the spirit is free from methylic alcohol; but if a copious precipitate of dark brown or black metallic silver separates, and the tube, after being rinsed out and filled with clean water, shows

a distinct film of silver, which appears brown by transmitted light (best seen by holding it against white paper), the spirit is methylated."

3. "A. Dupré has described (*THE ANALYST*, i. 4) the following method of detecting and approximately estimating the amount of methyl alcohol in spirituous liquids: Five ounces of the spirit are distilled twice, the liquid having been rendered alkaline the first and acid the second time, about two-thirds being passed over each time. The distillate is next shaken with dry potassium carbonate, and allowed to stand twelve hours. The upper layer is then removed with a pipette, and again twice distilled, about an ounce being driven over the first, and half an ounce the second time. This last half-ounce will contain any, methylic alcohol present in the original five ounces of the sample. All the distillations should be conducted in an apparatus having all the parts air-tight, expansion of the contained air being allowed for by a mercury valve. In this way the distillation can be effected without loss. About one-third of the last distillate is next diluted with about six times its measure of water, and in this spirituous liquid the alcohol is carefully determined, first by the density, and subsequently by oxidation to acetic acid, with estimation of the latter by titration with alkali. With pure alcohol, both methods should give results agreeing within 0.1 per cent. In presence of methyl alcohol, the oxidation process gives a sensibly lower result, as no fatty acid is formed by its oxidation. If any appreciable quantity of methyl alcohol be present, on opening the flask in which the oxidation is performed, a slight escape of gas will take place, owing to the carbon dioxide produced. With pure ethyl alcohol, on the contrary, a partial vacuum is formed. In a whisky to which 10 per cent. of methylated spirit was added, the specific gravity method gave 10.08 per cent. of alcohol in the diluted distillate, against 9.50 per cent. by the chromic acid method. A determination of the alcohol by Geissler's vaporimeter affords a useful check. Thus, the same whisky above mentioned gave 10.45 per cent. of alcohol by this process, owing to the presence of methyl alcohol increasing the tension of the vapour. The remainder of the distillate in which the methyl alcohol has been concentrated may be examined for that body by the tests described above."

Hehner has modified this process by measuring the quantity of chromate reduced, instead of the acetic acid produced, considering that much more decided indications could thus be obtained than by a method by which the most valuable constituent, the methyl alcohol, is really not estimated at all (*THE ANALYST*, xii. 25).

4. "The following process for testing alcohol depends on the presence of acetone, and was devised by J. E. Reynolds: 'Take 200 c.c. of the spirit, and rapidly distil off 50 c.c.; dilute the distillate with an equal volume of water, and slightly warm with addition of a few c.c. of solution of potassium hydrate. On cautious addition of mercuric chloride, the oxide at first thrown down is speedily redissolved; excess of the mercuric salt must be carefully avoided. The alkaline liquid should be filtered clear, much of the alcohol allowed to evaporate slowly, and the residue then divided in two portions. One part is to be violently boiled for a few minutes; a yellowish-white gelatinous precipitate will suddenly make its appearance if the acetone compound be present. In the second portion, dilute acetic acid, when added in excess, should produce a bulky, white, gelatinous precipitate, containing, when washed and completely dried, between 78 and 79 per cent. of mercury.'

5. "P. Cazeneuve distills 100 c.c. of the spirit, and collects each 10 c.c. of the distillate in separate cylinders. To each fraction he then adds 1 c.c. of a solution containing 5 grammes of potassium permanganate per litre. If the sample contained wood spirit, each fraction instantly reduces the permanganate with brown coloration, owing to the presence of acetone in all the distillates." If the alcohol be free from methyl compounds, but contains an appreciable quantity of aldehyde, the first two fractions will reduce the permanganate at once, but the following portions react less rapidly. This distinction is due to the low boiling point of aldehyde, causing it to become concentrated in the first portions of the distillate."

6. "From researches by M. Duclaux on the surface-tensions of the alcohols (*Annales Chim. et Phys.* (5), xiii. 76), it appears extremely probable that methyl alcohol could be detected, and even approximately determined, in spirituous liquids by simply noting the number of drops of the sample delivered by a pipette constructed to deliver 100 drops of water. A liquid containing 20 per cent. by volume of ethylic alcohol will give 176 drops, while methylic alcohol of the same strength will give only 147.5 drops."

A. Schwicker (*Chem. Zeit.*, xv., 1891, 914) states that powdered iodine acts upon a mixture of acetone and aqueous ammonia with the production of iodoform, and that alcohol gives no reaction under these circumstances, so that acetone can be detected in its presence. This reaction, which is described in *THE ANALYST* (xvi. 191), might perhaps be employed for the detection of methylated spirit.

These methods are either complicated, tedious, or uncertain, whilst some of them require comparatively large samples to be operated upon.

For the detection of acetone occurring in the acetonuria of diabetics, Le Nobel proposed the use of a solution of 5 grains of sodium nitroprusside in one ounce of water; when equal parts of this solution and the urine are mixed, and a few drops of ammonia added, a fine red colour gradually develops if there is any acetone, varying in depth according to the quantity present.

It is this reaction between acetone and sodium nitroprusside, in the presence of ammonia, which I proposed four years ago for the detection of methylated spirit; but I am now convinced that the reaction must be also due to some other constituent of wood spirit, because the colour is much more pronounced than it could be if it were caused by acetone alone. I have not yet been able to identify the constituent to which the colour is mainly due, but it is my intention to further investigate this point.

I use a 1 per cent. solution of the nitroprusside, which should be freshly prepared each time of use, mix equal parts of it and the sample, or distillate from the sample under examination, add a few drops of ammonia, and observe the colour after standing for ten or fifteen minutes, by which time the red colour will have become fully developed if acetone or some other constituent of wood spirit is present in appreciable quantity.

When examining spirituous liquids containing no solid matter in solution, the test may be applied directly, and in that case 3 or 4 c.c. of the sample is a quite sufficient quantity to take, 5 c.c. being a convenient amount. But in other cases it is necessary to distil, and in that case I take about 25 c.c. of the sample and test the first 5 c.c. of

the distillate, and occasionally two or three successive quantities of 5 c.c. ; or in very weak solutions it may be necessary to redistil the first 25 c.c. of the distillate from 100 c.c. of the sample.

With ethereal solutions we must distil to dryness in a water-bath, using a gentle heat at first and a higher temperature afterwards, and test several separate portions of the distillate, since the first parts, although they may give a brown or orange colour, do not show the reaction, and it is only towards the end of the distillation that the red colour will be observed, even when acetone or other constituent of wood spirit exists in the sample. It is best always to examine ether in this way, because in some samples containing no constituents of wood spirit a deep brownish colour may be produced by the test.

The advantages of this method are, the small quantity of the sample required, the rapidity and ease of execution of the operation, and the production of a distinct objective reaction.

The limits of the sensibility of the reaction must necessarily vary to some extent, since the wood spirit used for methylation is of variable composition. In one instance I was able to get the reaction in the distillate from an aqueous solution containing 4 per cent. of methylated spirit which had been reduced to the strength of rectified spirit, whilst I was unable to obtain it with a similar solution of another sample of methylated spirit ; but on distilling over 25 c.c. from 100 c.c. of that solution, and testing 5 c.c. of distillate from the latter, I got a strong red colour with nitroprusside. Dealing in a similar manner with a 2 per cent. solution of the same sample, reduced to the strength of rectified spirit, the reaction could be just observed, though perhaps not with absolute certainty.

When testing any fluid which is weak in alcohol, it is well to add 2 or 3 c.c. of strong pure alcohol to the distillate before mixing it with the nitroprusside solution, because an aqueous solution of that substance gives a yellow colour with ammonia, which is apt to mask a slight tinge of red, but which is prevented by the presence of a considerable quantity of alcohol.

The test will just reveal the presence of 1 per cent. of acetone in rectified spirit, and if the first 5 c.c. of a distillate from 25 c.c. are operated upon, 0.5 per cent. of acetone may be detected by it, and even 0.25 per cent. if three successive 5 c.c. of distillate are tested and compared with each other.

For the purpose of ascertaining the permanency of the reaction, in October, 1890, I put some caustic potash into a bottle of methylated spirit, and found that the distillate from the spirit still showed the reaction after 188 days. I did not try it again until exactly four years after adding the potash, and then I found that the reaction no longer succeeded.

Some methylated spirit heated with caustic potash under a reflux condenser for six and a half hours also failed to give the reaction.

The colour produced by the test must be an undoubted red if the presence of acetone or wood spirit is to be declared ; an orange or brown tint is not sufficient.

The red colour caused by the addition of nitroprusside to solutions of alkaline sulphides is of a different tint, and is formed immediately without requiring the

addition of ammonia; but if the presence of ammonium sulphide were suspected, it would be best to add some soda or potash to the sample before distilling.

Amongst other constituents of wood spirit, I find the nitroprusside reaction is produced by methyl alcohol which had been purified by calcium chloride, but not by methyl alcohol made from oil of wintergreen, nor by allylic alcohol; methyl acetate gives a faint purplish colour, but no distinct reaction with the test.

Furfural and acetal do not give the reaction; I have yet to ascertain which constituent is the chief cause of the colour reaction.

In order to determine whether methylated spirit invariably gives the reaction with nitroprusside, I asked for a sample from thirty-two methylators, and twenty-one of them have favoured me with twenty-five samples from England, Scotland, and Ireland, every one of which gave the reaction most distinctly, as did also various samples I have procured from retailers at different times, though of course the depth of colour in the samples has varied, because the composition of wood spirit is not constant.

I think, therefore, it is safe to assume that when the distillate from any alcoholic preparation gives a decided red colour with nitroprusside solution and ammonia within ten or fifteen minutes, methylated spirit is present; but in order to be able to assure myself that it would be safe to rely upon this reaction, I tried it with every preparation in the British Pharmacopœia which contains alcohol in any quantity whatever, or which is prepared from alcohol, over 160 in number, and in no instance, with two exceptions, was any red colour produced; whilst on again distilling the same preparations, after I had added some methylated spirit to them, I invariably succeeded in getting the reaction, as I did also with aconite, belladonna, soap, and compound camphor liniments made with methylated spirit, which preparations are now sanctioned by the Board of Inland Revenue; but a few preparations contain too little alcohol to admit of the identification of methylated spirit if present.

The exceptions to the trustworthiness of the test relate to paraldehyde and the collodium and collodium flexile of the Pharmacopœia. I have not found a red colour given by the test when applied directly to paraldehyde, but I have when it has been tried with distillates from that substance and several samples of collodium, although I could not be sure that pure alcohol or ether had not been used in their preparation; therefore, pending further investigation, I recommend that it should not be relied upon for testing those articles.

Nitroprusside gives no red colour with absolute alcohol, rectified and proof spirit, chloroform, chloroform made from methylated spirit, chloroform made from acetone, a sample of which I received from the late Mr. R. H. Davies, or pure ether; but it does with methylated ether, whilst with some commercial ethers it gives a brown colour, but those samples may be distinguished from methylated ether by applying the test to the distillate in the manner I have already described.

The test shows no reaction with spirit of nitrous ether made from rectified spirit, but it produces a red colour with some which I prepared from methylated spirit, and diluted with pure rectified spirit; and I obtained similar results with two samples of

it which I prepared from rectified spirit and from methylated spirit, in accordance with the formula of the London Pharmacopœia of 1851.

Three samples of potato spirit, which Dr. Vieth kindly procured in Germany for me, do not give the reaction, although one of them is of very inferior quality.

I have applied the test to distillates from brandy, whisky, gin, rum, and various kinds of wine, but in no instance was the reaction produced.

Methylated spirit now contains three-eighths of 1 per cent. by volume of mineral naphtha of a specific gravity of not less than 0.800, in addition to 10 per cent. of wood spirit, so it must be more difficult to substitute it for rectified or proof spirit in tinctures, etc., as a turbidity is produced on dilution with water. The detection of methylated spirit may, therefore, be an easier matter than it formerly was, as the distillates from it are rendered turbid by the addition of water, and the odour is perhaps more pronounced; but, on the other hand, the distillates from many pharmacopœial and other preparations become turbid on dilution with water, owing to the presence of volatile oils, and some of them possess an odour capable of masking that of the mineral naphtha, whilst it is still possible under some circumstances to procure unmineralized methylated spirit, which might possibly be used surreptitiously in the preparation of some articles; therefore, I think any reliable reaction for its detection must still be of some use.

If, in determining the substances to be used for the purpose of methylating, the Commissioners of Inland Revenue would insist upon the presence of a certain minimum amount of such of the constituents of wood spirit as most readily give the nitroprusside reaction, the detection of methylated spirit might be made more uniformly certain. It might, for instance, be stipulated that not only should 5 c.c. of all methylated spirit give a strong red colour with the test, but also that when 1 c.c. of the spirit, previously reduced to the strength of rectified spirit or to any other uniform strength, is made up to 25 c.c. with pure rectified spirit and distilled, the first 5 c.c. of the distillate shall, when tested with nitroprusside, give a red colour not less intense than that produced by the reagent in 5 c.c. of pure rectified spirit containing a certain percentage of acetone, or other constituent of wood spirit, to be decided upon after investigation.

I acknowledge with much pleasure my indebtedness to Dr. F. W. Stansfield, of Reading, for the help I have received from him in conducting the numerous experiments I have had to make for the purpose of this paper.

DISCUSSION.

The chairman (Mr. Hehner) said he thought that methods for the detection or estimation of methyl alcohol in alcoholic liquors should be divided into two classes—Continental and English, because Continental conditions were entirely different from those prevailing in England. In Germany and France methylation of spirits was not practised, and the methods of French and German chemists had for their object the determination of the exact amount of methyl alcohol in the ethyl alcohol used in commercial manufactures, as, for instance, of aniline dyes. It was very rare that this contained a large percentage of acetone, and therefore complicated methods were unavoidable. In England, on the other hand, pure wood spirit was hardly used at

all. The mistake was continually being made of comparing the methods without paying due regard to the differences in the conditions of their application, and as the expression "methylated spirit" was almost invariably understood by Continental chemists to mean "methyl alcohol," these misapprehensions had given rise to much confusion and contention. At the present time there was absolutely no definition of what was crude wood spirit. It must "stink" to a certain degree before it could be used, that was all, and the difficulty of defining a "stink" was so great that firms importing wood spirit were continually in conflict with the Customs. Whether or not Dr. Ashby's test would be adopted by the Customs, it was an exceedingly good one, and well deserved official recognition. Mr. Hehner mentioned a case occurring in his own practice which showed how desirable it would be that some such rule as that suggested by Dr. Ashby should be adopted. An importer of wood spirit mixed with alcohol the legal proportion of wood spirit, viz., at least 10 per cent., and exported the mixture to Australia, where it was tested, and found not to smell sufficiently; and although it already contained 10 per cent. of wood spirit, the Excise ordered it to be re-methylated with another 10 per cent. at the expense of the first methylator. He thought that the Society's very hearty thanks were due to Dr. Ashby for again laying his test before them in such a very detailed manner, and bringing forward such conclusive proofs as those he had exhibited before the meeting.

Dr. Dyer agreed with Mr. Hehner that the Society was very much indebted to Dr. Ashby. He had had some little experience with the Riche and Bardy test, having made several experiments with it, and had not succeeded in getting any ethylic alcohol which did not give the reaction indicative, or supposed to be indicative, of the presence of a small quantity of methyl alcohol. He would very much like to hear the experience of Mr. Allen, or any other member who had worked on the subject, which was a very important one, on account of the disputes which occasionally arose in Customs matters.

Mr. Allen congratulated Dr. Ashby on having brought this test to a practical issue. His own experience was not so large as Dr. Dyer seemed to suppose; he had never had occasion to come into conflict with the Inland Revenue officers in connection with any disputed cases, and had, therefore, not examined the published methods as critically as he might otherwise have done. The published methods were, he thought, complex or of doubtful accuracy, and he welcomed cordially this advance of Dr. Ashby's. There was one point about which he sought information, which could probably be supplied by a word from Dr. Ashby. Sweet spirits of nitre, when prepared according to the London Pharmacopœia directions, contained a good deal of aldehyde, and the orange-yellow reaction it gave with sodium nitroprusside would, he thought, be likely to interfere with the distinctness of the acetone reaction upon which Dr. Ashby's test depended.

Dr. Ashby exhibited the test applied to one portion of rectified spirit containing 2 per cent. of acetone, and to another containing 2 per cent. of aldehyde.

Mr. Allen said he thought it possible that sweet spirits of nitre made with methylated spirit would contain not more than 1 per cent. of acetone, while containing

as much as 3 per cent. of aldehyde, and in such a case he should not expect the acetone reaction to be distinct. He thought that analysts were indebted to Dr. Ashby for bringing forward his test, which, if found satisfactory in the hands of others, would certainly come into general use. Nothing found its level so readily as an analytical process, and the methods at present used in this connection were anything but satisfactory.

Dr. Ashby said there was no danger whatever of the aldehyde reaction interfering with that of the acetone. For instance, the distillate from spirits of nitrous ether made with rectified spirit gave a very faint orange colour—in fact, only a slight deepening of the colour of the nitroprusside—but the distillate from spirits of nitrous ether made with methylated spirit by himself, diluted down in the ordinary way with rectified (not methylated) spirit, gave a very distinct red reaction. The difference between two ethers, one methylated and the other not methylated, was very marked and could not be mistaken; with many perfectly pure ethers no colour reaction at all was obtained.

Mr. Cribb then read the following paper :

THE NEED FOR FULLER STATISTICS OF ADULTERATION.

BY CECIL HOWARD CRIBB, B.Sc. (LOND.).

I RECENTLY wished to obtain an approximate but, as far as could be reliable, minimum estimate of the total loss to the country owing to adulteration, but, after looking into the matter a little, found the task to be an impossible one. There is no great difficulty in ascertaining with very fair accuracy the annual consumption of most articles of food. In the case of all duty-paying articles and of all imported ones, the consumption may be found in the reports of the Inland Revenue and Customs Departments and of the Board of Trade. With regard to home produce, not paying duty, valuable, and I think trustworthy, information is to be obtained from the journals of the Royal Agricultural Society and of the Statistical Society, various trade papers, and elsewhere. It is, however, with the adulteration side of the question that the difficulties begin.

As the only body to which copies of the quarterly reports of all the public analysts are sent, the Local Government Board might reasonably be expected to give the needed statistics in their reports; but those who know those somewhat tardy annuals will know that no such information is to be found in them. The percentage of samples found to be adulterated is given, but the nature and average extent of the adulteration is not stated. This being the case, I sent out, as the only other method of getting first-hand information, a printed letter to those of the County Councils which are most active as regards the adulteration question, asking for copies of their public analysts' reports for the previous year. As would be anticipated, the reports received varied enormously, hardly two were constructed on the same plan; and while some analysts barely complied with the Act, by stating only the number of samples analysed and the number found adulterated, others gave detailed information as to every case of adulteration, including not only the results of legal proceedings, but also much

other matter, often of great interest and of high educational value for the authorities for whom they were written.

It certainly seems strange that after nearly twenty years, during which the Sale of Food and Drugs Acts have been working, no uniform system of recording the results of our labours has been adopted ; and at the present juncture it is extremely important that some serious attempt should be made to place before the public an estimate, based on the best available data, of the national loss owing to the evil of which it is our duty to aid in the suppression. Of its magnitude, the majority of people have not the remotest idea, and in consequence there is no doubt that in many cases we are not yet taken seriously by the public, and that the authorities who appoint us often do so reluctantly—perhaps only at the instigation of the Local Government Board—and attach a very second-rate importance to our work.

Under these circumstances it seemed worth while to bring the whole matter before the Society ; and my main object now is to lay before you a suggestion which, if carried out, will, I think, not only result in a large addition to our knowledge as regards the prevalence of adulteration, its effect on commerce and on the food bill of the country, but will greatly increase the prestige of the Society, and tend to gradually establish it in the minds of the public at large as *the* body, and the only body, competent to express an opinion on the scientific aspects of the question, and will cause it to be generally recognized as the *source* of all authoritative information on the subject.

My suggestion is that the Society should collect statistics of the work of all public analysts in England and Wales, or better still in the United Kingdom, and should issue a comprehensive report based thereon—not a red-tape report, made up of dry and valueless tables, but one, if possible, dealing with the larger aspects of the question, with the loss caused by adulteration, and its relation to the expenditure on the suppression of adulteration, to the total expenditure on food, and to the total national income ; with the effect of adulteration (if any) on health, on commerce, and on our commercial relations with other countries, stating not only the percentage of samples found adulterated, but the nature and average extent of each kind of adulteration ; mentioning new offences, new tricks of the trade, new methods of evading the law, and all the High Court decisions which may have been given during the year.

This is, of course, a very ambitious programme, and it might be years before it could be carried out in its entirety ; but it may, perhaps should, be regarded as more an expression of what we want than of what we are likely to get, but it is at all events at what we should aim. Many other matters which have been overlooked might doubtless be suggested, but those mentioned will serve, at all events, as a basis for discussion. The matter is not one to be lightly entered into, or without due thought and preparation ; but in the belief that it is worthy of serious consideration, I propose to put briefly before you a few of the advantages which would, I think, follow the execution of the design, and then say a word or two as to methods of collecting the information required.

To commence with the least selfish reasons, such a report as has been suggested should surely be of great value, as well as of considerable interest, to the general public.

About four and a half million of our population are in some way engaged in the preparation, production, or sale of food—everyone has a personal and private interest in the matter; and surely the people who gloat over the death rate, or get excited over the census, or the number of stamps sold by the Post Office, will spare a portion of their attention for a matter of at least equal interest and importance.

Moreover, it is high time that the whole question received some share of the popular attention, in order that the many extraordinary delusions at present current on the subject should be cleared away; and for this purpose it might perhaps be possible to issue an abridged and less technical report than that intended for experts. To County Councillors and vestrymen, and all engaged in the working of the "Adulteration" Acts, the utility of such a report is obvious.

It is, however, to members of this Society individually, as well as to the Society as a whole, that this scheme should be of the most use.

There are many points of professional interest on which, for various reasons, we require information. No one, I believe, outside the Local Government Board knows even the names of those holding the 237 appointments as public analysts which up to the present have been made, much less their qualifications, the conditions on which they are appointed, and the salaries they are paid. I am not prepared to advocate the *publication* of all such details, but I certainly think they should be accessible to every member of this Society, and should be tabulated and arranged ready for production before any future Select Committee or Royal Commission.

Again, if the statistics we require are collected in the way I suggest, it would give the Society an excellent opportunity, without practically any further trouble or expense, of getting information on any points of special scientific interest or importance that may crop up during the year, such as the occurrence of abnormal milks and butters, the employment of boracic acid and other antiseptics in butter and milk. We should also probably be enabled to get by exchange copies of the annual reports of the Health Departments of various foreign Governments, such as those of the Municipal Laboratory of Paris, of the German Reichsgesundheitsamt, and the excellent bulletins of the Inland Revenue Departments of Canada, and various American States, which, if present at all, are very difficult to find in the British Museum, and, with the exception of a few copies in the library of the Chemical Society, are hardly likely to be met with in any other library in London. Some of these reports are of great value and interest, and it is much to be regretted that they should be at present inaccessible to most, if not all, of us.

It can hardly be doubted that the prestige of the Society as a whole would be greatly increased by the execution of the suggested scheme. Anyone looking at the minutes of the proceedings of the present Select Committee on Adulteration can hardly fail to be struck by the fact that the first witnesses examined were for the most part not public analysts at all, but farmers, milk dealers, butter merchants, grocers, clerks from the Local Government Board, and the Government beer and tobacco examiners; and though no doubt public analysts will have their turn, it must be admitted that we should have cut a much better figure before the public eye had we, in our *corporate* capacity, been in possession of the information which Local Government Board representative retailed at second hand.

Further, by issuing a report of its own, the Society cannot fail to get recognized

Finally, what is perhaps the most difficult question remains—who is to do the work? Assuming that all the members of the Society are willing to do their part by obtaining and furnishing the information needed, who is to compile and edit the report? It would, of course, be unfair to add to the burden already resting on the shoulders of the secretaries, beyond perhaps asking them to distribute the printed forms, and the actual compilation must of necessity fall into other hands. I trust, however, that if this is the only obstacle in the way of the adoption of the scheme, it will not fail on that account, and I can but express my own willingness to aid in its execution to the utmost of my power.

DISCUSSION.

Mr. Allen said he was afraid that the realization of Mr. Cribb's suggestions would take a considerable time to effect. Something of the sort in an imperfect form used to be published in *THE ANALYST*, but since the death of Mr. Wigner this had fallen through. The unsatisfactory nature of the statistics already available was in some measure the result of the Local Government Board's instructions regarding the form in which reports should be made. Some public analysts assumed that the barest compliance with these instructions was sufficient to constitute a report, but he had always looked upon the official form rather as an appendix to a more detailed report, in which suggestions might be made as to the best mode of dealing with any special points which happened to arise. In the West Riding he was required to make an annual report, as well as a quarterly report, and years ago he began giving a very comprehensive table of results, the compilation of which now required an enormous amount of labour owing to the expansion that had occurred as time went on, and the difficulty in collecting the information which it was necessary to obtain from inspectors in remote parts of the district, some of whom he had never seen. This was particularly the case with regard to information as to the results of legal proceedings, and he pointed out that, if there was so large an expenditure of time and trouble involved in the making of one such report as he spoke of (which was compiled for the use of the County Council only), the collection of the elaborate statistical information which would be necessary for the practical and efficient carrying out of Mr. Cribb's ideas would, in the case of many public analysts, take up so much of their time as to render it doubtful whether they would be able to undertake it. He thought it would be an advantage to print in *THE ANALYST* a full list of the public analysts at present acting, giving the districts for which they acted, as information on this head was now only obtainable from the bluebook, and this was inaccurate. He thought Mr. Cribb deserved their thanks for bringing the matter forward, but the amount of work that would be involved in the practical carrying out of his suggestions was very considerable, and they could not ask the secretaries to undertake any more than they already had to deal with.

Mr. Richmond said that such a report as Mr. Cribb suggested would undoubtedly be read with great profit by a large number of the general public, though he did not think that it would be much improvement on that of the Local Government Board, which, although a little tardy and somewhat complicated, contained all that was

really required, including details as to convictions and the total amount of the fines imposed. This report was compiled from official sources, and thus would naturally have advantages over the purely voluntary one proposed. All public analysts in extensive practice must have felt practically certain that a considerable number of their samples were adulterated, but the adulteration not being sufficiently marked to justify an adverse report, these samples had to be passed as genuine; in fact, practically all cases of scientific adulteration would be included among the genuine samples. Then, again, differences, sometimes immense, between the standards of different places, the greater or less experience and zeal of the inspectors who collected the samples, and other causes of a like nature, would affect the proportion of samples returned as adulterated (apart from the quality of the articles themselves), and so tend to depreciate the value of this huge collection of figures, which, too, would almost certainly be incomplete, as some few at least out of the 280 public analysts would be sure to fail to report. It was very doubtful, seeing that a really complete and accurate report could not be drawn, whether it would be worth while to spend a considerable amount of time and money upon it.

Mr. Hehner said that although the desirability of some such scheme of records was beyond question, its accomplishment presented many difficulties, and it seemed to him that it ought to form part of a great scheme for the reorganization of the Sale of Food and Drugs Act. It could not be done by the secretaries, as was shown by the experience of Mr. Wigner and Mr. Heisch years ago, and he thought it would never be satisfactorily done until it was undertaken by the State. Perfection, of course, was not attained quickly, but all possible progress towards it should be made. The statistics at present available were very insufficient, and did not represent the actual state of adulteration and the consequent loss to the country at all, nor did they show in what way the country was benefited by the public analysts. It was impossible to gather whether certain forms of adulteration had died out, or whether there was any decrease in the average proportions of foreign ingredients now added, which he was certain was the case in milk, at least.

Mr. Cribb said he was sorry to see that, while everybody seemed to agree as to the desirableness of the scheme, nobody seemed to be anxious to do anything. As to who was to do the work, he would be the last to expect the secretaries to undertake it, although he thought that even they would not object to sending out the forms along with their other notices—which would give them practically no more trouble than at present. He would have liked to see the matter referred to the Council, or to a committee. He would be very glad to do his best, provided that the statistics were obtained, to collate them. The thing could, however, only be carried out satisfactorily under the ægis of the Society. At all events, a trial might be made, say for a year.

Mr. Allen asked if Mr. Cribb proposed to collect statistics from all public analysts, including those who were not members of the Society.

Mr. Cribb said he thought there would be no great difficulty in that, if the non-members were approached in the proper way.

Dr. Dyer remarked that Mr. Cribb was not a county analyst, and could,

perhaps, hardly appreciate the difficulties which county analysts would have in furnishing or being furnished with all the statistical information suggested. They had one central authority in a County Council, but this often comprised virtually a number of all but separate authorities in the local police division. An analyst who, like Mr. Cribb, was attached only to a Metropolitan parish, had all his fellow-officers within a short distance, and could easily ask for and obtain information difficult to obtain over the scattered area of a whole county.

Mr. Cribb said that his chief desire that evening was only to initiate the matter, and that it should not be allowed to drop. If it were referred to the Council or to a committee, perhaps the Society might see its way to taking some steps on the lines he had indicated.

Mr. Bevan said that it was only fair that the matter should go before the Council, as Mr. Cribb had taken a good deal of trouble and time over it. There were several things that he personally could not approve of, but Mr. Cribb himself was not finally decided as to details. Dr. Dyer and he would bring the matter before the Council at their next meeting.

Mr. Hehner said that it might be possible for the Council to try such a scheme, and to appoint a committee with that object.

Mr. Bevan exhibited two novel pieces of apparatus, illustrations and descriptions of which will appear in our next issue.

In the absence of the author, Dr. Dyer read a paper on "Roasted Chicory," by E. G. Clayton, the publication of which is unavoidably postponed.

The Resemblance between the Reactions of the Alkaloids and of Acetanilide.
E. Schär. (*Arch. Pharm.*, cccxxii., 249, through *Chem. Zeit.*)—Tafel has pointed out that anilides, such as acetanilide, give a reaction with sulphuric acid and an oxidizing agent which resembles the strychnine reaction. Flückiger has drawn attention to a similarity between the reaction of morphine with sulphuric acid, containing nitric acid, and that of acetanilide with the same reagent. Schär has tested both these statements; he finds, with respect to the strychnine reaction, that this differs in two main points from that yielded by acetanilide. (1) The play of colour shown by strychnine is from blue to methyl violet, whereas that exhibited by acetanilide is rather a blue-purple-red coloration. (2) The introduction of the oxidizing agent into the solution of strychnine in sulphuric acid induces a deep violet colour, which gradually changes—through cherry-red, purple-red, and blood-red—to yellow-red, whilst in the case of acetanilide, there is a rapid change from purple-red, through violet-red, into a dirty blue-green, olive-green, or brown-green.

The reaction of sulphuric acid, containing nitric acid, on morphine and on acetanilide is certainly very similar; but acetanilide gives no reaction with sulphuric acid which contains selenic acid, titanous acid, molybdic acid, or tungstic acid, and thus should not be mistaken for morphine. Furthermore, morphine gives a deep

red-brown colour with sulphuric acid and bismuth subnitrate, whilst acetanilide gives a dark yellow colour, becoming carmine-red at the edges of the mass.

A. G. B.

A Simple Method of Purifying Commercial Ether. M. Ekenberg. (*Chem. Zeit.*, xviii., 1242.)—For most analytical purposes, sufficiently pure ether can be easily obtained from the commercial article by mixing it with from 5 to 10 per cent. of its volume of a liquid paraffin, which boils above 300° C., and distilling at 40° to 50° C. The paraffin retains the alcohol and oxidation products in the retort, and if much water be present, this will form a layer beneath the paraffin. By heating the paraffin to 120° C., the impurities are expelled, and the oil rendered fit for further use. The method is quite efficacious in removing acids, evil-smelling compounds, and peroxides, and may also be applied for the purification of light petroleum, aldehyde, chloroform, etc.

A. G. B.

Utilization of Sodium Peroxide in Analysis. O. Kassner. (*Arch. Pharm.*, ccxxxii., 226-240.)—The oxygen which is evolved from sodium peroxide in water contains ozone, a fact which probably accounts for the activity of the peroxide as an oxidizing agent.

Häussermann has shown that the addition of sodium peroxide to the solution of a chromium salt precipitates chromium hydroxide, which, at the ordinary temperature, is redissolved, and oxidized to sodium chromate; at low temperatures a brown solution of sodium perchromate, $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 28\text{H}_2\text{O}$, is produced.

When sodium peroxide is added to a solution of uranyl nitrate the usual yellow precipitate of sodium uranate, $\text{Na}_2\text{U}_2\text{O}_7$, is formed, but is immediately redissolved; the solution gives a yellow crystalline precipitate of sodium peruranate, $\text{Na}_4\text{U}_2\text{O}_8 \cdot 8\text{H}_2\text{O}$, when alcohol is added. If the solution be warmed before the alcohol is added, it becomes red, and alcohol then precipitates a red oil, which speedily becomes crystalline, and proves to be the mixed uranate, $\text{Na}_2\text{U}_2\text{O}_8 \cdot \text{UO}_2$, discovered by Fairley.

Manganese is precipitated as hydrated peroxide when sodium peroxide is added to its solutions, but further oxidation is impossible, because permanganates are reduced by the peroxide. Similarly, iron is precipitated as ferric oxide, no ferrite or ferrate being formed.

Clark (*Trans. Chem. Soc.*, 1893, 1079) has already dealt with the separation of chromium from manganese and iron by the aid of sodium peroxide. The author claims that the results obtained by such a method are not low, as stated by Clark; thus, by adding sodium peroxide by degrees to a mixture of solutions of chrome alum and manganese sulphate, heating for some time, and washing and weighing the manganese peroxide as usual, 99.96 per cent. of the manganese was estimated, whilst 99.87 per cent. of the chromium was found by reducing the filtrate and precipitating with ammonia. By a similar procedure with a mixture of ammonium ferrous sulphate and chrome alum, 99.93 per cent. of the chromium and 99.81 per cent. of the iron were estimated.

Potassium ferricyanide is reduced to ferrocyanide by sodium peroxide, and since

the ferrocyanide can be titrated by potassium permanganate in an acid solution, the following method may be adopted for the analysis of red prussiate: The weighed sample (0.5 gramme) is dissolved in about 100 c.c. of water, and a little sodium peroxide (0.06 gramme) is added; the solution, thus decolourized, is heated until all effervescence has ceased, acidified with dilute sulphuric acid, diluted with water, and titrated with potassium permanganate solution. A slight green precipitate may be formed by the acidification, but since this is only due to the iron oxide in the sodium peroxide, and reduces as much permanganate as the ferrocyanide reduces, it may be disregarded.

Sodium peroxide precipitates the black sesquioxide of cobalt from solutions of that metal, but only the green hydroxide from solutions of nickel. Mercury, silver, and gold are precipitated in the metallic state from the solutions of their salts by sodium peroxide; but solutions of platinic chloride and palladous chloride in hydrochloric acid are not so reduced, apparently because the double sodium salts which are first formed are stable. When the hydrochloric acid is removed by silver nitrate, both salts are reduced.

Separation and Estimation of Antimony, Tin and Arsenic.—The mixed sulphides are stirred with about 30 c.c. of cold water in a tall beaker, and sodium peroxide is added little by little until a small portion gives no coloured precipitate on acidification. The contents of the beaker are now transferred to a silver crucible, and evaporated to dryness; the residue is kept in fusion for some time, and then digested with water containing one-third of its volume of alcohol. The sodium pyroantimonate is filtered off, washed, and weighed as antimonyl antimonate in the usual manner. When the alcohol has been evaporated from the filtrate, dilute sulphuric acid is added until the liquid is feebly acid, the precipitated stannic acid is redissolved by caustic soda, an excess being avoided, and carbon dioxide is passed through the solution until it becomes turbid. By now adding ammonium chloride, heating for half an hour, and leaving the solution at rest for nearly twenty-four hours, all the stannic oxide is precipitated, and may be weighed as usual. To precipitate the arsenic, which exists in the filtrate as sodium arsenate, ammonia and magnesia mixture are added; after being allowed to remain for forty-eight hours in the cold, the ammonium magnesium arsenate is filtered, washed with ammonia, and weighed as magnesium pyroarsenate.

The figures quoted show that, in two analyses conducted in this manner, 99.86 per cent. of antimony, 99.91 per cent. of tin, and 99.75 per cent. of arsenic were recovered in the first case, and 99.67 per cent. of antimony, 99.64 per cent. of tin, and 99.83 per cent. of arsenic, in the second case.

The author claims that by this method the time of oxidation of the sulphides is reduced to a minimum (half to one and a half minutes), and that tin will not so perpetually accompany the antimony as is the case in the usual method. Moreover, if a qualitative test for arsenic in Marsh's test be required, there is no nitric acid to be expelled before this can be performed.

For qualitative testing, the mixed sulphides are oxidized as described above, and a small portion of the solution (after it has been thoroughly boiled to decompose the excess of peroxide) is added to some acidified potassium iodide solution; a liberation

of iodine will show the presence of antimony. If this be present, the main solution is heated for some time, alcohol is added, and the pyroantimonate is filtered off. After the alcohol has been evaporated from the filtrate, this is made feebly acid with sulphuric acid and ammonium chloride is added; the precipitated stannic oxide is filtered off, and arsenic sought in the filtrate. A. G. B.

Comparative Tests of different sorts of Glass with reference to their Chemical Composition. By F. Foerster. (*Zeit. für Analyt. Chemie*, 1894, Viertes Heft, pp. 381-396.)—The author gives tables of the composition of seventeen kinds of glass used for chemical apparatus, and of the manner in which they resist the attack of water, hot and cold, and of caustic alkalies. The two which offered the most resistance when treated with water at 20° for eight days, and at 80° for three hours were specimens of Schott's Jena glass, and had the following percentage composition:

	K ₂ O	Na ₂ O	CaO	ZnO	MnO	Al ₂ O ₃ + Fe ₂ O ₃	SiO ₂	B ₂ O ₃
1.	—	11	—	—	0.05	5.0	71.95	12.0
2.	—	9.8	7.0	5.0	0.3	3.5	74.4	—

No. 1 was far more readily attacked when boiled with caustic soda for three hours than were vessels made of the best ordinary glass, and is, therefore, not suitable for analyses where caustic alkalies are used. No. 2 offered about the same resistance to alkalies that good ordinary glass does, but was far less attacked by hot and cold water. On the other hand, it is difficult to manufacture chemical apparatus from it. The author alludes to another borate glass from the same manufactory which has not these disadvantages, but gives no particulars of it in his tables. C. A. M.

Contributions to the Analysis of Lard. G. Halphen. (*Journ. de Pharm. et de Chimie*, 1894, xxx., pp. 241-247).—The application of the silver nitrate test to the fatty acids instead of the fresh lard is recommended. Filtration removes substances from pure lard fatty acids which reduce silver nitrate, and thus one of the objections brought against Becchi's test is avoided. For the detection of foreign animal fat, as well as cotton-seed oil, the author, in collaboration with M. Bishop, has devised a process for taking separately the iodine numbers of the liquid and solid fatty acids in the lard.

Sear's method of separation is used. 10 grammes of the fatty acids are dissolved in 200 c.c. of CS₂ in a 250 c.c. flask. To the solution 5 grammes of ZnO are added, the flask well corked and shaken at intervals for six hours. The soluble zinc salts are separated from the insoluble by filtration. The filter is well washed with CS₂, the washings being added to the soluble salts in a tared flask. The CS₂ is distilled off, and the residue dried for an hour at 90°, while a current of dry air is passed over them. The increase of weight shows the quantity of zinc salts furnished by the liquid part of the fatty acids. To estimate the combined zinc 50 c.c. of normal H₂SO₄ are added, with constant shaking, until all the acids are liberated. These remain liquid at from 18° to 20°. The liquid in the flask, excluding the layer of fatty acid, is made up to 200 c.c. Rather more than 100 c.c. are removed with a pipette and filtered. 100 c.c. of the clear filtrate are then titrated with normal

soda, from the result of which the weight of zinc combined with 100 grammes of the fatty acid can be calculated. This is also an index of the molecular equivalent of the acids. The iodine number is determined on the acids direct.

The zinc salts insoluble in CS_2 left on the filter are decomposed by boiling with HCl , and their iodine number taken. It is noteworthy that the soluble zinc salts obtained from pure lard, beef suet, and mutton suet, are of an amber colour, while those derived from cotton-seed oil acids or from lards adulterated with cotton-seed oil are orange-red.

The following table gives the results of analyses.

Nature of Fat.	Iodine No. total Acids per cent.	Iodine No. Liquid Acids per cent.	Iodine No. Solid Acids per cent.	Soluble Zinc Salts.		Liquid acids per cent. grammes.	Solid acids per cent. grammes.	Index of Saponification in ZnO per cent.
				Weight. Grms.	Colour.			
Pure American lard ...	63.5	91.95	28.83	64.7	amber	57.14	42.86	14.7
Pure French lard ...	59.0	86.61	32.91	57.83	amber	50.72	49.28	15.8
American fat (A) ...	83.56	117.1	47.58	61.2	orange	53.86	46.14	15.4
American fat (B) ...	83.56	114.04	48.56	63.06	orange	55.6	44.4	15.2
Beef suet pressed ...	17.01	75.60	12.95	7.84	amber	6.8	93.2	15.2
Mutton suet	37.84	80.26	17.52	35.2	amber	31.44	68.56	14.5
Cotton-seed oil	02.361	129.03	49.53	78.15	orange	69.06	30.96	14.9

A further communication on the subject of mixtures is promised.

C. A. M.

EXTRACTS FROM THE EVIDENCE GIVEN BEFORE THE SELECT COMMITTEE ON FOOD PRODUCTS ADULTERATION, ON JULY 11, 18 AND 25.

MR. RICHARD BANNISTER.

(Continued from page 264.)

7. EVIDENCE REFERRING TO VINEGAR.

786. Do you think that a pure malt-vinegar is not made from simple malt?—In the vinegar trade they have followed the same lines as the brewers. At one time brewers were compelled to make beer from malt only, and then they were allowed to use malt and grain, or malt and sugars; the vinegar brewers have gone in the same line.

1795. On the question of malt-vinegar, do you agree that when malt-vinegar is asked for, it should be vinegar distilled from malt?—No.

1796. Made from malt?—Made from malt and grain; made from malt, or malt and grain.

1797. But you would not give any further latitude in the making of vinegar; or rather, when malt-vinegar is demanded, you would consider that it should be made

of malt, or malt and grain?—I should liberally read the expression “from grain,” because in some cases it has been said that the word “grain” refers to barley. If a man uses other grain, or other starchy matter, I should consider that that brewed with malt was malt-vinegar.

1831. You referred to malt-vinegar, and you said that it had to be made of malt, or malt and grain; but in using the term “malt,” do you mean necessarily malt made from barley?—No; it would include any malt.

1832. Malt may be fairly made from any wholesome grain?—Yes.

1833. And that would be included in your term “malt”?—Yes; but, as a rule, you find that nearly all malt is made from barley.

1834. But you would not necessarily require that it should be made from barley?—Certainly not.

8. EVIDENCE REFERRING TO GINGER.

2700. Have you ever found that spent ginger has been used as an adulterant to any extent?—I have had samples that I have examined that did contain spent ginger.

2701. Is there any proper commercial use for the product that is known as spent ginger?—No, unless a certain quantity of extractive matter is left in the ginger.

2702. Would the sale to the public of genuine ginger with which spent ginger had been mixed be a fraud on the purchaser?—It would depend upon the price, because the price of ginger varies greatly.

9. EVIDENCE REFERRING TO LARD.

2885. In your opinion, is the use of a moderate quantity of beef-stearine—say 10 per cent.—for the purpose of stiffening lard an adulteration?—At home, in the summer, we always used to put a small quantity of mutton-suet into the lard to keep it hard, and make it convenient to work for domestic purposes. . . .

10. EVIDENCE REFERRING TO SUGAR.

2686. If coloured or yellow crystals are sold as Demerara sugar, ought the sellers, in your opinion, to be prosecuted under the Sale of Food and Drugs Act for adulteration, or under the Merchandise Marks Act for giving a false trade description?—It strikes me that it should be under the Merchandise Marks Act, so long as the colouring-matter is so small that it does not amount to adulteration.

2687. You do not consider that the colouring process is an adulteration except beyond a certain point?—I have examined a sample that has been coloured, and the amount of adulteration by colouring is so small, that it could not be considered adulteration.

2688. Where do you think adulteration begins?—It is very difficult to say where one begins and the other ends; it depends entirely upon the article itself.

11. EVIDENCE REFERRING TO STANDARDS AND LIMITS.

829. In connection with that subject, I should like to know your opinion as to the desirability of extending the system of standards on even recognised limits. You have a legally-enacted limit in the case of spirits, have you not?—Yes.

850. What I wish is the opinion of yourself, as a member of a very important

department, as to the possibility of extending this system of known standards so that the analysts in different parts of the country might work on something like the same lines?—As far as the analysts are concerned, there is no doubt that the establishment of standards would certainly bring in unity, whereas sometimes there is discord at the present time—there is no doubt about that.

851. And they do make standards for themselves, and then you make other standards for your department, and that sometimes brings you into conflict?—Yes.

852. Is it your opinion that whereas the standards of your department, or of a central department, would be official, such a standard set up by a central official department would be more convenient and more just on the whole than a standard which may vary with the taste of each individual?—There is no doubt that it would be easier to do.

853. It is proposed, as you are aware, that there should be some central department.—I have seen the statement in the draft Bill.

854. Assuming that such a central department were set up, do you think that there would be any difficulty in at all events very largely extending the list of articles in which a recognised standard might be published?—I think there would be considerable difficulty. There is no doubt that, so far as the establishment of standards is concerned, if the standard were equal, it would be a great deal easier to work the Adulteration Act so far as the analysts are concerned. But it is quite another question for the producers; they would have something to say to that, too.

855. But do you not think it is desirable to apply the same principles, so far as possible, to all classes or articles?—Evidently, as far as you can, as long as they do not conflict with other interests.

856. Then what I ask you is, whether you have considered that the same principles as are applied to spirits and to milk and butter (that is to say, the fixed definitions of standard) could not be extended to a number of other products?—Certainly it could.

885. On the policy of these points we should not regard you, of course, as an expert witness; but there is a point on which you can give us better information, I think, probably, than anyone else, and that is about the proposal that the Chemical Court of Appeal should be changed from the Inland Revenue Department to some other department, such as the Local Government Board. You are aware that that proposal has been seriously made by the analysts?—Yes.

886. And that they express themselves as very much dissatisfied with the present system, and say that the arrangements for the Chemical Court of Appeal should be entirely remodelled? I am glad to see that you have their proposition before you.—It was given to me the last time that I was here.

887. Would you describe in your own words from the statement what has taken place?—In the first place, we have not got an Inland Revenue Chemical Department at the present time, because there has been a committee of inquiry into the different chemical works of the Government; and now we are a Government Laboratory made up of the Customs Department and the Inland Revenue Department, and have to take over the whole of the work from the other departments. With regard to the suggested Board of Reference, the suggestion is that “there shall be appointed a

board or committee, consisting of the chief chemical officer of the Inland Revenue Laboratory, a person nominated by the General Medical Council, three persons, being public analysts, nominated by the Local Government Board, and a person nominated by the Board of Agriculture." If you notice about the Board of Reference, in all the trades that are concerned, there is not a reference to those trades at all; in fact, when we look at the numbers, you will find that there are to be three public analysts, and there are to be three others.

888. So that, in the first place, you object to that proposal of a Board of Reference as an unfair composition?—Yes; it is really a reference in which, so far as the trades are concerned, they have no voice in the matter. The reference seems to me all wrong.

889. Have you anything to say on the proposal for transferring the department from the position in which it now stands to the Board of Trade or the Local Government Board?—In regard to that, I think that the first point which has to be considered is whether the Board of Inland Revenue, as referees under the Act, have done their duty, or whether they have not. If you look at the work which it has done, and the decisions that have been arrived at by the magistrates, I think you must confess that they have done their duty.

890. What I should like you to explain to the committee is the opinion of your department on the subject. I understand you to say emphatically that there is no necessity for any change?—Not the slightest.

891. And you would deprecate being put under any other department than that under which you serve at the present time?—I feel in this way. Of course, the Government of the day is the authority that I have to obey, and if the Government of the day were to say that certain things had to be done, I, as a loyal civil servant, should try to carry out their instructions to the utmost of my ability.

892. Then you consider it a question of policy, and not a question upon which you, from your official position, can express an opinion?—I think I can express an opinion.

893. And that opinion would be hostile to that suggestion altogether?—Certainly.

894. As a matter of fact, I suppose that there would be no particular difficulty in a transfer of the department from the position in which it now stands to another department?—We do the Board of Trade work now at the present time, the whole of it, and have done it for years.

895. But, as regards the Local Government Board Department, you are not in very close communication with them, apparently. You do not report to them at all, do you?—Whenever any question arises on which we have to report to the Local Government Board Department, we do report to them, but nothing beyond that.

896. I mean in connection with the administration of the Sale of Food and Drugs Act?—If there is any question that is necessary to be referred between the two departments, it is referred, but as a rule it is not necessary.

1798. Many suggestions have been made, I believe, on the part of the Society of Public Analysts, that there should be a central department having the power relegated to them that Somerset House now enjoys, consisting of six or more, or

less, perhaps, gentlemen of scientific standing, including three public analysts, who should work out methods of analysis and lay down limits and standards; do you agree with that suggestion?—I do not.

1799. I think you raised the point that the commercial interests should be represented?—Certainly. As soon as that paper was read, the very first suggestion made by a chemist and druggist was that there should be two chemists and druggists put on; you can easily see, therefore, that if you have a central board of that kind, you must have the trades represented. But I think you want a more central laboratory to do the work.

1835. Now, if a public analyst writes to you for a standard for an article, do you give it?—We give him a limit; any information that we can give him on the subject, we do give him.

1836. That is in the ordinary course of things?—Yes.

1837. Do you think these standards or limits would be well fixed, as has been suggested, by a board of analysts and scientific men?—Personally, I am opposed to standards altogether. I think that we can do a great deal better without them.

1838. If any board of analysts and other scientific people were arranged for helping the working of the Act, do you think that that, irrespective of standards, would be desirable?—I think it would not, unless everybody was represented—the scientific part and the trading part.

1839. This is the question which I wanted to come to: perhaps the analysts would not assent to that?—I expect they would not; I do not know.

1840. You think that if there is a board, the traders should in some way be represented upon it as well as the scientific element?—Certainly.

(Concluded.)

NOTE.—These extracts from the evidence of Mr. R. Bannister were compiled from the official transcript of the shorthand notes issued after each sitting of the committee. In the Blue-book which has been subsequently issued certain alterations will be met with.

REVIEW.

ADULTERATION (AGRICULTURAL FERTILIZERS AND FEEDING STUFFS). By FRANCIS H. CRIPPS-DAY, M.A. (Cantab), of the Middle Temple and Oxford Circuit, Barrister-at-Law. (London: Stevens and Sons, Limited. Price 5s.)

The author of this book disclaims in the preface any intention to produce a "complete manual upon the agricultural, mercantile, scientific, and legal sides of the subject," but seeks to introduce only "so much of each as will serve to put the farmer, the merchant, the analyst, and the lawyer in possession of a succinct introduction to any of the subjects foreign to his own profession."

In the introductory chapter the author gives a sketchy, but perhaps a sufficiently lengthy, description of the principles underlying the scientific application of fertilizers and feeding stuffs, which in the main is not calculated to seriously mislead the general reader. In the following chapter the author attempts to classify systematically the manures and feeding stuffs found in the market, but the attempt has not been very

successful. "Superphosphates (natural)" and "ammonium sulphates prepared from sewage" are described as "artificial organic fertilizers," while it is solemnly asserted that superphosphate, prepared from coprolites, is "commonly adulterated with gypsum (formed in the process)." The list of feeding stuffs omits any mention of such well-known substances as earth-nut, palm-nut, and coconut cakes, but includes (possibly cattle may like a "relish" at breakfast!) such substances as "fish and flesh," "herring-meal," etc.

In chapter iii. the full text of the Fertilizers and Feeding Stuff Act, 1893, is given, with notes by the author. What the exact legal value of these may be we are not in a position to decide, but some of the definitions are worth quoting. Thus, after considering the meaning of the word "fertilizer," the author offers a definition of his own: "A *fertilizer* is anything which, when applied to the soil, enters wholly or in part into the composition of the vegetable produce of the said soil. Under this definition *lime is a fertilizer, gypsum is not.*" (The italics are ours.) Again, "suitable for feeding purposes" is defined, "If the feeding stuff contains any deleterious substance, or is in a condition whereby it is unwholesome to feed cattle upon, it will not be fit for feeding purposes." It is also stated that "the frauds in the sale of fertilizers by means of dyes are not touched by the Act."

Chapter iv. is devoted to "The Analytic Evidence." It will probably be news to those analysts appointed under the Act to learn that, when called as a witness, "The district analyst *must* be asked what education he has undergone fitting him to give an expert opinion. Diplomas of Universities enable a chemist to certify as to chemical analysis, or a botanist as to certain microscopical experiments, but only a specialist in agricultural science is in a position to give the opinion as required in Forms A and C."

Pages 55-72 are occupied with a more or less accurate *resumé* of the processes used by analysts in examining fertilizers and feeding stuffs, but as the information given is in many cases wrong, and in all cases perfectly worthless to the chemist and quite unintelligible to the layman, it is difficult to understand what good can result from its publication. Interspersed with these methods are descriptions of adulterations which, in many instances, are calculated to mislead. Thus, "In the tricalcic phosphate lime is present to the amount of 38.7 per cent., whereas in basic phosphate the percentage of lime is 43.7." "Mineral superphosphate implies a minimum of 17 per cent. soluble phosphate." "The presence of . . . sand (in guano) will sustain a prosecution." It will perhaps be news to the author to learn that good genuine Peruvian guano as now imported rarely contains less than 10 per cent. of sand, and often much more. "Special tests will be made to discover what is the percentage of gelatine, if guaranteed, as is the case sometimes with North American (Cotton) cakes, it being a substance of especially nutritive value for calves." This might be necessary if cotton cake was a suitable food for calves, if it contained gelatine, and if it was the custom to guarantee in these cakes substances which are known not to be present.

The remainder of the book is concerned with translations of the French, Belgium, and German laws on the subject, and with abstracts of the United States' law, which will doubtless prove useful for reference.

A. S.

The Analyst,

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INDEX OF SUBJECTS.

A.	PAGE		PAGE
Acetic acid in vinegar, Estimation of, A. R. LEECH	224	Asphaltene, Definition of	41
Acetone, Detection of water in, SCHWEITZER & LUNGWITZ	234	Asphalt question, The, S. F. PECKHAM	63
Acetyl numbers of lard, cotton-seed-oil, and tallow, E. SPAETH	252	Asphaltum, On the technical analysis of, L. A. LINTON	41
Acids, volatile fatty, Mathematical deductions from the distillation of, H. D. RICHMOND	193	Austrian alimentary code, Extract from (Wine)	117
Tables of the rates of distillation of	196, 197		
Air, Estimation of dust in, K. ARENS	137	B.	
Alkaloids and glucosidal bodies, Quantitative separation and chemical characteristics of, in forensic cases, C. KIPPENBERGER	201, 204	Badouin's reaction for the detection of sesame-oil in olive-oil, E. CARLINFANTI	222
Methods of estimation of single	203	Barium thiosulphate for standardizing iodine solution, PLIMPTON & CHORLEY	111
Separation of mixtures of	204	hydroxide solution in carbon determination in steel, J. O. HANDY	191
Acidimetric estimation of vegetable, L. F. KEBLER	278	Beer, The detection of fluorine in, HEFELMANN & MANN	185
Aluminium, amalgamated, as a reducing agent, WISLICIENUS & KAUFMANN	213	Estimation of glycerin in, A. PARTHEIL	251
Analysis, volumetric, Barium thiosulphate for standardizing iodine solution in, PLIMPTON & CHORLEY	111	Detection and estimation of glycerin in, MOLHAUT	223
Ang-khak, a Chinese dye used for coloring articles of food, H. C. PRINSEN	198	Benzene, Estimation of thiophen in, E. DENIGES	188
Annual Address of the President	25	Blood-stains, Detection of, in forensic cases, F. GANTTER	186
Annual Meeting of the Society of Public Analysts		Bone-black, Rapid and accurate analysis of, W. D. HORNE	69
Anthracene, Estimation of paraffin in crude, HEUSLER & HERD	142	Boric acid, Estimation of, J. N. DUPASQUIR	223
Antimony as antimonious antimoniate, Estimation of, O. BRUNK	190	Boron, Estimation of, H. MOISSAN	22
arsenic, and tin, Separation and determination of, W. HAMPE	46	Bromine in the presence of chlorine, Estimation of, W. WENSE	139
Antipyrine and thalleioquin reaction, The, J. DUNCOMMUN	234	Burette, An improved, F. OETTEL	238
Apparatus for preserving and delivering standard solutions, J. C. CHORLEY	15	Butter analysis, contribution to (Refractometer), J. DELAITE	59
constant level for distilling flask, J. C. CHORLEY	16	Butter, an abnormal, J. SAMUELSON	221
APPOINTMENT:—		Examination of, E. POLENSKE	277
Mr. G. R. Thompson to Monmouthshire	264	and margarine, test for distinguishing, J. ROLFFS	221
Apples, Estimation of zinc in desiccated, L. LEGLER	251	Viscosimetric examination of, C. KILLING	66, 94
Arachis-oil, Modification of Renard's process for the determination of, H. KREIS	136	Viscosimetric examination of, for foreign fats, N. WENDER	220
Arsenic in iron and steel, Volumetric estimation of, A. MIGNOT	235		
Separation of, from other elements by methyl alcohol and hydrochloric acid, FRIEDHEIM & MICHAELIS	235	C.	
Detection of, in presence of selenium, L. DAWIDOW	113	Cadmium and copper, New method for the estimation of, A. S. CUSHMAN	214
Toxicological detection and estimation of, E. BARILLOT	22	zinc, and nickel, Separation of copper from, P. N. RIKOWA	23
		Calcium oxide in quicklime, Method of determining, STONE & SCHEUCH	19
		Solubility of, in sucrose solutions	19
		Californian red wines, Colouring matter in, W. D. BIGELOW	112
		Candle material, Determination of melting-point and composition of	40
		Carbohydrates in milk, Identification and estimation of, A. W. BLYTH	121

	PAGE		PAGE
Carbon estimation in steel, Superiority of barium hydroxide solution in, J. O. HANDY ...	191	Cream, average amount of fat in, during 1894 ...	55
„ in iron, N. W. method of estimating, L. SCHNEIDER ...	65	D.	
Carbonic acid, Examination of liquid, L. GRÜNHUT ...	113	Dextrin, Estimation of ...	226
Castor-oil, Anomalous behaviour of, towards Hübl solution ...	51	Diastase, Chemical nature of, T. B. OSBORNE ...	232
Cellulose, Quantitative estimation of, G. LANGE ...	283	„ can be salted out with ammonium sulphate or magnesium sulphate ...	233
Charcoal, Rapid analysis of animal, W. D. HORNE ...	69	Duclaux's method for the estimation of "volatile fatty acids," the laws governing volatility deduced therefrom, and their application to analysis, especially to that of butter, H. D. RICHMOND ...	193, 217
Cheese, Contribution to the study of the ash of, MARIANI & TASSELLI ...	168	Dust in the air, Estimation of, K. ARENS ...	137
„ Note on two samples of "filled or lard," R. BODMER ...	268	E.	
Cheeses, Composition of some English, CHATTAWAY, PEARMAIN & MOOR ...	132	Elaidin reaction, Influence of temperature on, A. P. LIDOW ...	178
Chicory, Roasted, E. G. CLAYTON ...	12	F.	
Chlorine in presence of iodine, Detection of, P. N. RAIKOW ...	47	Fats, Gravimetric determination of bromine absorption in, O. HEHRER ...	49
„ and iodine, Quantitative separation of, P. N. RAIKOW ...	47	„ and hydrocarbons, A new method of analysing, M. CRISMER ...	209
„ in the presence of bromine, Estimation of, W. WENSE ...	139	„ Critical temperatures of dissolution ...	210
Chloroplatinates, Volumetric analysis of, L. L. DE KONINCK ...	140	Fatty acids, Separation of solid and liquid, E. TWITCHELL ...	165
Chromium in chrome ore, Method of determining, E. CLARK ...	141	„ Note upon Muter's, Jean's, Rose's, methods of separation of ...	165, 166
Cinnamon, Notes on, DYER & GILBARD ...	129	„ Viscosity increases with molecular weight ...	220
„ Table of analyses of ...	130	„ „ free in oil-cakes and other feeding stuffs, DYER & GILBARD ...	241
„ Microscopic appearance of ...	131	„ bodies, Action of sulphur on unsaturated, J. ALTSCHUL ...	256
Citric acid, Characteristic reaction of, L. STAHR ...	188	„ Critical temperatures of dissolution, a new constant for their examination, L. CRISMER ...	257
Cocanut-oil in lard compounds, E. B. KENRICK ...	96	Fluorine in wine, Detection of, NIVIERE & HUBERT ...	185
Coffee, A new alkaloid in, PALADINO ...	141	„ beer, Detection of, HEFELMANN & MANN ...	185
„ beans, On the fat, sugar, and tannin of, HEFELDT & STUTZER ...	249	Fodders, Estimation of starch in ...	19
„ Note on an adulterated sample, PEARMAIN & MOOR ...	176	„ Estimation of woody fibre in, A. P. AITKEN ...	35
Cola-nut, On the chemical composition of, UFFELMANN & BÖMER ...	42	„ Importance of removing oil before determining the fibre ...	36
Confectionery, On the composition of some vegetable coloring matters for use in, G. POSETTO ...	188	Formaldehyde, Methods for the detection of ...	154
Copaiba, Detection of oil and gurgun balsam in, E. HIRSCHORN ...	252	„ Action of, on albumin, 156, 157, 167	
Copper, The wet assay of, R. S. DULIN ...	164	Formalin as a preservative of milk samples, E. J. BEVAN ...	152
„ salts, Methods of analysis of, L. SOSTEGNI ...	189	„ Note on the detection of, RICHMOND & BOSELEY ...	154
„ bullion, Estimation of tellurium in, C. WHITEHEAD ...	189	„ as a milk preservative, S. RIDEAL ...	157
„ and cadmium, New method for the separation of, A. S. CUSHMAN ...	214	„ Action of, on food stuffs, WEIGLE & MERKEL ...	167
„ Separation of, from cadmium, zinc, and nickel, P. N. RAIKOW ...	23	Formic acid, Determination of, with potassium permanganate, H. C. JONES ...	207
CORRESPONDENCE:—		„ „ Estimation of, F. FREYER ...	208
Cocanut-oil in lard compounds, E. B. KENRICK ...	96	Fustic, Interpretation of the results of the analysis of, C. S. BOYER ...	208
Letter from Dr. Dupré ...	144		
„ „ Dr. E. Frankland ...	192		
„ „ Mr. W. C. Young ...	216		
Cotton-oil in lard, Note on the detection of, E. B. KENRICK ...	136		

INDEX.

	PAGE		PAGE
G.		Iron and steel, Volumetric estimation of	
Gelatin rendered insoluble by formaldehyde	44	arsenic in, A. MIGNOT	235
Geranium-oil in rose-oil, On the detection of,		„ and nickel, Separation of, CAMPBELL	116
R. JEDERMANN	48	AND ANDREWS	116
Glass, Action of magnesium mixture on,		K.	
L. L. DE KONINCK	198	Kjeldahl-Jodblauer-Gunning method, Note	
Glucosazone, Solubility of	125	on	40
„ can be readily shaken out with		„ process, Uselessness of, for deter-	
ether	127	mination of nitrogen in platino-	
Glucose, A new method of estimating, by		chlorides, DELEPHINE	111
ammoniacal copper solution, ALLEIN &		„ method for the determination of	
GAUD	21	nitrogen, B. DYER	252
Glue and glue-yielding substances, The		„ process, Arrangement for conduct-	
analysis of, W. FAHRION	254	ing	253
Glycerin, Crystallized, E. J. BEVAN	181	„ process, Modification of, for sub-	
Glycerol in wines, Estimation of, MANCUSO-		stances containing nitrates	253
LIMA & SGARLATA	183		
„ beer, Detection and estimation		L.	
of, M. MOLHAUT	223	Lactosazone, Solubility of	125
„ wine and beer, Estimation of,		Lard, Note on a sample of supposed, B.	
A. PARTHEIL	251	KIRTO	32
Gold estimation by parting, Influence of the		„ Note on detection of cotton-oil in,	
metals of the platinum group on, E.		E. B. KENRICK	136
PRIWOZNIKI	262	„ Examination of, A. GOSKE	222
Gunning method for estimating total nitro-		„ Analysis of, M. MANSFIELD	222
gen in fertilizers, W. E. GARRIGUES	39	Lard analysis, The phosphomolybdic test as	
		applied to, G. F. TENNILLE	63
H.		„ Results of a number of tests	
Hide-powder, Action of alcoholic soda on...	178	in	
Honey-dew, The composition of, and the		Larderine, Note on a sample of, R. BODMER	33
influence of a summer rich in honey-dew		Lathyrus sativus, Feeding experiments with	
on the condition of honey, E. VON RAUMER	16	Indian peas containing,	
Honey, Water in, J. GRAFTIAN	251	J. HUGHES	169
Horseflesh, Chemical recognition of, W.		„ Microscopic structure of,	172
NIEBEL	252	Lead and lead ores, Analysis of, NISSENSON	
„ Chemical process for recog-		& NEUMANN	286
nising, M. HUMBERT	95	Leather, Analysis of chamois	180
Hubl's method of iodine absorption, J.		„ Analysis of glacé kid	180
FEHRAIM	176	„ Determination of sulphuric acid in,	
„ iodine process, Theory of, and sug-		P. F. JEAN	62
gested improvement, WALLER	280	Linseed-oil, Examination of raw and boiled,	
Hydrobromic acid, Formation of, in oil		H. AMSEL	70
testing not due to presence of water	50	„ Examination of raw and boiled,	
Hydrocarbons and fats, A new method of		F. FÜLSINGER	137
analysing, M. CRISMER	209	Liquid carbonic acid, Examination of, L.	
Hydrogen peroxide, The concentration and		GRÜNHUT	113
distillation of, R.		Litmus solution, Preparation of, W. SCHAFER	37
WOLFFENSTEIN	38	Luteol, A new indicator, W. AUTENRIETH	188
„ „ in green plants, Detec-			
tion of, A. BACH	112	M.	
I.		Mace, Chemical variations of different kinds	
Indicators in volumetric analysis, use of		of, E. SPAETH	200
litmus and methyl orange, H. LUNGE	65	Magnesia mixture, Action of upon glass,	
Indigotin, New method of estimating, J.		L. L. DE KONINCK	138
SCHNEIDER	211	Magnesian pyrophosphate in the citrate	
Iodine, Curious behaviour of unsaturated		method, How to obtain pure white, H.	
fatty acids with	52	NEUBAUER	20
„ in organic substances of the fatty		Maize as an adulterant of oatmeal, J. WHITE	30
series, M. C. SCHUYTEN	234	Maltose, Estimation of	226
„ and chlorine, Quantitative separa-		„ Rotatory power of, H. OST	253
tion of, P. N. RAIKOW	47	„ Specific gravity of solutions of,	259
Ionone and irone, Detection and estimation		Manures, Determination of potash in, W. E.	
of, TIEMANN & KRÜGER	234	GARRIGUES	66
Iron, New method of estimating carbon in,		Martius' yellow in macaroni, etc., Detection	
L. SCHNEIDER	65	of, F. SCHAFER	225

	PAGE
Maumené's test for oils, H. D. RICHMOND	58
Meat-extract, Estimation of gelatin and albumin in, E. BECKMANN	44
Meat-extracts, Comparative examination of the constitution of, A. STUTZER	182
Valuation of	182
Meat-extracts and commercial peptones, Analysis of the nitrogenous constituents of, A. STUTZER	246
Microbes found in water, List of	97
Milk analysis, Acetic acid does not completely remove proteids in	128
Contribution to, E. BECKMANN	42
Employment of dehydrated sodium sulphate in	20
Estimation of fat, E. BECKMANN	42
Estimation of solids-not-fat by freezing	43
Inversion of cane-sugar by citric acid	138
Method of clearing whey for polarimeter	121
Use of Mohr-Westphal balance in, C. H. WOLFF	115
Milk, Apparent increase in total solids under the influence of formalin	152
Case involving a sample of abnormal, R. BODMER	265
Composition of, and the conditions affecting it, as shown by Dr. Bell's analyses, J. F. LIVERSEGE	7
condensed, The composition and analysis of, PEARMAIN & MOOR	265
Detection of watered by examination of milk serum, LESCOEUR	200
Identification and estimation of carbohydrates in, A. W. BLYTH	121
Formalin as a preservative in, S. RIDEAL	157
Necessity of adding preservatives to while fresh	156
Note on commercial condensed, A. H. ALLEN	274
New formula for estimating composition of from total solids and fat	57
New method of estimating fat in, FERNANDEZ-KRUG & HAMPE	20
Recknagel's phenomenon in, H. D. RICHMOND	1
Milks, condensed, Tables of the analyses of	271, 275
Milk and milk products, Composition and analysis of, during 1894, H. D. RICHMOND	54
Milk samples, Use of formalin as a preservative of, E. J. BEVAN	152
Milk scale, Correction for Richmond's	58
Milk-sugar, Estimation of by Gerrard's cupric-cyanide process	124, 127
Estimation of by phenylhydrazine	125
Rübner's test for	95
Morphine, Fallacies of post-mortem tests for	88

	PAGE
N.	
Naphthol, Detection of, H. SPINETTE	211
Nickel and iron, Separation of, CAMPBELL & ANDREWS	116
Nickel, cadmium and zinc, Separation of copper from, P. N. RIKOWA	23
Nitrates, Determination of nitrogen in, M. KRÜGER	45
Nitrites in presence of iron salts, Detection of, H. KRAL	24
Nitrocellulose, Estimation of nitrogen in, D. STANOJEWITSCH	21
Nitrogen in fertilizers, On the Gunning method for estimating total, W. E. GARRIGUES	39
Nitrogen in nitrates, nitro-compounds, and nitroso-compounds, Determination of, M. KRÜGER	45
O.	
Oatmeal adulterated with barley	32
Oatmeal, Note on maize as an adulterant of, J. WHITE	30
Obituary notice, Dr. W. Morgan, of Swansea Officers and Council for 1895	30
Oil analysis, Attempt to use heat evolved by sulphur chloride in	148
Relation between Hübl number and rise in temperature with bromine	149
Oil, Arachis, modification of Renard's process for the detection of, H. KREIS	136
cotton-seed, Becchi's test for	222
cakes and other feeding stuffs, Free fatty acids in, DYER & GILBARD	241
olive, Iodine chloride as a substitute for Hübl's solution	177
Examination of	45
On Hübl's method of iodine absorption, J. EPHRAIM	176
raw and boiled linseed, Examination of, H. AMSELL	70
Linseed, Note on the iodine and bromine absorptions of, R. WILLIAMS	276
Sesame in olive, Baudouin's reaction, E. CARLINFANTI	222
Oils, A new thermal method for the examination of, HEHNER & MITCHELL	146
Chloro-iodine, Numbers of certain	138
Colour reactions of olive and others, NEGRI & FABRIS	45
Detection of albuminous matters in	178
Gravimetric determination of bromine absorption in, O. HEHNER	49
Maumené's test for, H. D. RICHMOND	58
Relation between the Hübl and Maumené figures	147
Tables of the iodine and bromine numbers of various	51
Stannic bromide test for	175
Testing of, with the refractometer, T. H. PEARMAIN	134
P.	
Paraffin in crude anthracene, Estimation of, HEUSLER & HERDE	142

	PAGE
Peas, Indian, containing lathyrus sativus, Feeding experiments with, J. HUGHES	169
" Microscopic structure of	172
Pepper, The examination of, W. BUSSE	180
" Valuation of, by means of coloring matter in husk	181
Peptone, Estimation of gelatin and albumin in, E. BECKMANN	44
Peptones, Analysis of commercial	246
" Note on the adulteration of commercial, M. L. HOGOUNENG	94
Petrolene, Definition of	41
Petroleum, Estimation of sulphur in, F. HEUSLER	187
Perchromic acid, Solvents for, W. M. GROSVENOR	113
Phenylhydrazine, Employment of, in estimation of dextrose, levulose, and saccharose, LINTNER & KRÖBER	167
Phosphomolybdic acid test in lard analysis, G. F. TENNILLE	63
Phosphoric acid, Estimation of, by titration of the molybdate precipitate, B. W. KILGORE	17
" " Method of obtaining pure white magnesium pyrophosphate in the estimation of, H. NEUBAUER	20
" " The determination of, H. PEMBERTON	95
" " Simple method of determining by molybdate solution, J. HANAMANN	115
" " Estimation of "available," in Thomas slag, G. SANI	189
" " Estimation of "citrate-soluble," in Thomas slag, P. WAGNER	215
Platino-chlorides, Uselessness of Kjeldahl's method for determining nitrogen in, DELEPHINE	111
Poirvrette in pepper, Philoroglucol test for	181
Potash in manures, Determination of, W. A. GARRIGUES	66
Potash determination in fertilizers, Addition of calcium chloride to solution in, R. DE ROOPE	66
Potassium nitrate, Removal of perchlorate from, V. PANAOTOVIE	23
Q.	
Quicklime, Method of determining calcium oxide in, W. E. STONE & F. C. SOEHOCH	19
Quinine, The micro-chemical examination of, H. BEHRNS	92
R.	
Recknagel's phenomenon, Note on, H. D. RICHMOND	
Refractometer, Testing of oils with, T. H. PEARMAN	134
Refractometers, Difference in scales of	135
Resin in soap, Gladding's process for the determination of, L. ARCHBUTT	6

REVIEWS:—

	PAGE
Aids to the analysis of food and drugs, PEARMAN & MOOR	262
Air, water, and disinfectants, C. M. AIKMAN	249
Chemical analysis of oils, fats, and waxes, and of the commercial products derived therefrom, BENEDIKT & LEWKOWITSCH	119
Chemistry of urine, a practical guide to the analytical examination of diabetic, gouty, and albuminous urine, A. H. ALLEN	263
Poisons, their effects and detection, A. W. BLYTH	239
Science and art of bread-making, W. JAGO	238
The Sale of Food and Drugs Acts, BELL & SCRIVENER	24
Rose-oil, On the detection of geranium-oil in, R. JEDERMANN	48
Resin in soap, Examination of the Twitchell method of determining, EVANS & BLACK	60
S.	
Salicylic acid in wine, Detection of, M. SPICA	184
Saltpetre, The purification of, from perchlorate, V. PANAOTOVIE	23
Sandal-wood-oil, Note on the adulteration of, PEARMAN & MOOR	174
Selenium, Detection of arsenic in presence of, L. DAWDOW	113
Sewers, Absence of typhoid bacteria in	106
Silicates, Lead carbonate as an agent for opening up, P. JANNASCH	191
Silk from cotton, To distinguish	179
Slag, Thomas, Estimation of phosphoric acid in, G. SANI	189
" " Estimation of phosphoric acid in, P. WAGNER	215
Soap, Determination of resin in, by Gladding's process, L. ARCHBUTT	6
Soaps, Examination of the Twitchell method of determining resin in, EVANS & BLACK	60
Soda solution, Action of alcoholic, on albumin and glue-yielding substances, W. FAHRION	178
Sodium peroxide, Note on the analysis of, L. ARCHBUTT	3
Starch, A comparison of the methods for the determination of, W. E. STONE	17
" Solution, The preservation of, H. KRALL	23
" Rapid estimation of, P. L. HIBBARD	64
" New method of estimating, by iodine, DENNSTADT & VOITGLANDER	210
" Determination of, H. OST	226
" Estimation in brewers' grains and forage material	19
" Microscopical distinction between maize and oatmeal	31
Streams, Purification of by green plants, bacteria, etc., BOKONYI & DUCLAUX	91
Sugar Estimation by ammoniacal cupric sulphate solution, ALLEN & GAUD	21

	PAGE		PAGE
Sugar Estimation, Action of alkaline copper solutions on sugars, KJELDAHL ...	227	Thiophen in benzene, Estimation of, G. DENIGES ...	188
" " Influence of air, and of quantity of alkali and tartrate in ...	227	Tin, arsenic, and antimony, Separation and determination of, W. HAMPE ...	46
" " Kjeldahl's tables for glucose, fructose, invert, galactose, lactose, and maltose ...	228, 229, 230	Tobacco, Method of estimating ammonia in, in presence of nicotine, V. VERDRÖDI ...	255
" " Peska's tables for glucose, invert, lactose, and maltose ...	231	V.	
" " Method of converting cuprous oxide into cupric, K. FARNSTEINER ...	258	Varnishes, The analysis of, P. C. McILHINEY ...	93
" " By means of potassium copper carbonate, H. OST ...	259	" Processes used in oil analysis unsuitable for that of ...	194
" " Tables of various sugars for use with Ost's solution ...	260	Vinegar, Estimation of acetic acid in, A. R. LEEDS ...	224
" " Volumetric, by ammoniacal copper solution, Z. PESKA ...	230	Viscosimetric examination of butter, C. KILLING ...	66, 94
" Products of the action of Fehling's solution on ...	230	Volumetric analysis, Use of litmus and methyl-orange in, G. LUNGE ...	65
" Solutions, Preparation of for polarimeter, STIFT & PETZIWAL ...	232	" " Standardization of sulphuric acid, F. S. SHIVER ...	143
" Invert, Influence of lead acetate on the determination of, BORNTRAGER ...	232	W.	
Sugars, Estimation of dextrose, levulose, and saccharose, LINTNER & KRÖBER ...	167	Walnut shells, Microscopic appearance of ...	131
Sulphocyanic acid, On a method of estimating, M. T. GOUDOIN ...	43	Water of springs containing zinc, W. F. HILLEBRAND ...	17
Sulphur, On the detection of small quantities of metals of the second group in precipitated, R. FRESSENIUS ...	45	" Chemical and bacteriological examination of, with A. Dupré's remarks on the 1893 fever epidemic at Worthing ...	73
" in organic volatile compounds, Determination of, C. F. MAXBURY ...	61	" Organic matters undergoing putrefaction in absence of air do not yield nitrates ...	83
" petroleum, Estimation of, F. HEUSLER ...	187	" of several deep wells, Nitric nitrogen of ...	84
" Action of, on unsaturated fatty bodies, J. ALTSCHUL ...	256	" in which no bacteria could be found, cause of epidemics ...	98
Sulphuric acid, Standardization of, S. F. SHIVER ...	143	" Number of micro-organisms present in, no criterion of purity ...	100
" " in leather, Determination of, P. F. JEAN ...	62	" Comparison of the organic carbon and nitrogen results of the Metropolitan Water Companies ...	159
Sulphuretted hydrogen in analysis, Thiocetic acid as a substitute for, SCHIFF & TARUGI ...	62	" analysis, Erratic results of the Frankland and Armstrong process of ...	73, 160
T.		" " chemical, What does it teach? ...	74
Tannin, Experiments on, L. FAJANS ...	256	" " Unreliability of bacteriological methods in ...	75
" Determination of, by metallic oxides, W. H. KRCG ...	283	" " Failure of bacteriological examination in detecting impure water ...	79
Tellurium in copper bullion, C. WHITEHEAD ...	189	" " Importance of knowing source of water in ...	80
Thiacetic acid, Action of, on metallic solutions, N. TARUGI ...	213	" " Indications afforded by nitrates ...	82
" " Preparation of ...	214	" " Indications afforded by nitrates, ammonia, phosphates, and organic matter in ...	85, 86
" " as a substitute for hydrogen sulphide in analysis, SCHIFF & TARUGI ...	62	" at Worthing, Extract from Dr. T. Thompson's report on ...	77
		" filtration, Bacteriological analysis a good guide for judging of the efficiency of ...	26

	PAGE		PAGE
Waters, Analysis of the London, for the years 1891, 1892, 1893 ...	161	Wines, Colouring matters in Californian red, W. D. BIGELOW ...	112
„ causing epidemics, Table of analyses of ...	103	Woody fibre in fodders, Estimation of, A. P. AITKEN ...	35
Westphal balance, Use of, in milk analysis, C. H. WOLFF ...	115	Wool from cotton, To distinguish ...	179
Wine, Extract from the Austrian alimentary code ...	117	Woollen, pseudo-, fabrics, E. G. CLAYTON ...	174
„ Determination of glycerol, and estimation of man- nitol in, MAN- CUSO - LIMA & SGARLATA ...	183	Worthing, Remarks on the fever epidemic of 1893, A. DUPRÉ ...	73
„ „ salicylic acid in, M. SPICA ...	184		
„ „ tannin in, A. VIGNA ...	184	Z.	
„ „ fluorine in, NI- VIERE & HUBERT ...	185	Zinc-bearing spring waters, W. F. HILLK- BRAND ...	17
„ Estimation of potassium sulphate in, L. HUGOUNENG ...	184	„ cadmium, and nickel, Separation of copper from, P. N. RIKOWA ...	23
„ „ glycerin in, A. PARTHEIL ...	251	„ in desiccated apples, Estimation of, L. LEGLER ...	251
		„ Note on the determination of, W. F. SHIMER ...	148
		„ Preparation of pure, MYLIUS & FROMM ...	236
		„ Volumetric determination, and appli- cation of a new indicator, G. C. STONE ...	236

INDEX OF AUTHORS' NAMES.

	PAGE		PAGE
A.		BODMER, R., Note on a sample of lardering ...	33
AITKEN, A. F., The determination of woody fibre in fodders ...	35	„ Note on a recent case involv- ing a sample of abnormal milk ...	265
ALLEN & GAUD, New method of estimating glucose by alkaline copper solution ...	21	„ Note on two samples of "filled" or "lard" cheese ...	268
ALLEN, A. H., Note on commercial condensed milks ...	274	BOKORNY & DUCLAUX, Purification of streams by green plants, by bacteria, and other- wise ...	91
ALTSCHUL, J., The action of sulphur on un- saturated fatty bodies ...	256	BORNTRÄGER, The influence of plumbic acetate on the determination of invert sugar ...	232
AMSEL, H., On the examination of linseed- oil and boiled linseed-oil ...	70	BOYER, C. S., Interpretation of some results in the analysis of some extracts of fustic ...	208
ARCHBUTT, L., Note on the analysis of sodium peroxide ...	3	BRUNK, O., The estimation of antimony as antimonious antimoniate ...	190
„ Note on Gladding's process for the determination of resin in soap ...	6	BUSSE, W., The examination of pepper ...	180
ARENS, K., Estimation of dust in air ...	137		
AUTENRIETH, W., Luteol, a new indicator ...	188	C.	
B.		CAMERON, C. A., Note on unusual specimens of milk ...	111
BACH, A., The determination of hydrogen peroxide in green plants ...	112	CAMPBELL & ANDREWS, Separation of nickel and iron ...	116
BARILLOT, E., Toxicological detection and estimation of arsenic ...	22	CARLINEANTI, E., Badouin's reaction for the detection of sesame-oil in olive-oil ...	222
BECKMANN, K., Contribution to milk analysis „ Estimation of gelatin and albumin in peptone ...	44	CHATTAWAY, PEARMAIN & MOOR, The com- position of some English cheeses ...	132
BEHRENS, H., On the microchemical exami- nation of quinine ...	92	CHORLEY, J. C., Apparatus for preserving and delivering standard solutions ...	15
BEVAN, E. J., Crystallized glycerin ...	181	CHORLEY, J. C., Constant level apparatus ...	16
„ The use of formalin as a pre- servative of milk samples ...	152	CLARK, E., Method of determining chro- mium in chrome ore ...	141
BIGELOW, W. D., Colouring matter in Cali- fornian red wines ...	112	CLAYTON, E. G., Pseudo-woollen fabrics ...	174
BLYTH, A. W., The identification and esti- mation of carbohydrates in milk ...	121	CRISMER, M., A new method of analysing fats and hydrocarbons ...	209

INDEX.

	PAGE		PAGE
CRISMER, L., Critical temperatures of dissolution, a new constant for the examination of fatty bodies	257	G.	
CUSHMAN, A. S., A new method for the separation of copper and cadmium in qualitative analysis	214	GANTTER, F., The detection of blood-stains in forensic cases	186
D.		GARRIGUES, W. E., On the Gunning method for total nitrogen estimation in fertilizers	86
DAVOLL, D. L., junr., Fallacies of post-mortem tests for morphine	38	" The determination of melting-points and the composition of some candle material	40
DAWYDOW, L., The detection of arsenic in the presence of selenium	113	" The determination of potash in manures	66
DELAITE, J., Contribution to butter analysis (Abbé refractometer)	59	GOUDON, M. T., On the method of estimating sulphocyanic acid	43
DELEPHINE, Usel-ness of Kjeldahl's method for determining nitrogen in platino-chlorides	111	GRAFTIAN, J., Water in honey	251
DENIGES, G., Examination of medicaments containing iodine	186	GOSKE, A., The examination of lard	222
" Examination of thiophen in benzene	188	GROSVENOR, W. M., jun., Solvents for perchloric acid	113
DENNSTEDT & VOIGTLÄNDER, A new method for the quantitative estimation of starch	210	GRÜNHUT, L., Examination of liquid carbonic acid	113
DULIN, R. S., The wet assay of copper	164	H.	
DUPRÉ, A., Note on the chemical and bacteriological examination of water, with some remarks on the fever epidemic at Worthing in 1893	73	HAMPE, W., The separation and determination of arsenic, antimony, and tin	46
DUCOMMUN, J., Antipyrine and thalioquin reaction	234	HANAMANN, J., Simplified method for determining phosphoric acid by means of molybdate solution	115
DYER & GILBARD, Notes on cinnamon	129	HANDY, J. O., The superiority of barium hydrate solution as an absorbent in carbon determination in steel	191
" Free fatty acids in oil-cakes and other feeding stuffs	241	HEFELMANN & MANN, Detection of fluorine in beer	185
DYER, B., Kjeldahl's method for the determination of nitrogen	252	HEINER, O., On the determination of the bromine absorption of fats gravimetrically	49
E.		HEINER & MITCHELL, A new thermal method for the examination of oils	146
EPHRAIM, J., On the Hübl method of iodine absorption	176	HERVELDT & STUTZER, On the fat, sugar, and tannin in coffee-beans	249
EVANS & BLACK, An examination of the Twitchell method for the determination of rosin in soap	60	HEUSLER & HERDER, The estimation of paraffin in crude anthracene	142
F.		HEUSLER, F., On the estimation of sulphur in petroleum	187
FAHRION, W., Action of alcoholic sodium hydrate solution on albumin and glue-yielding substances	178	HIBBARD, P. L., Rapid estimation of starch	64
" The analysis of glue and glue-yielding substances	254	HILLERAND, E., Zinc-bearing spring waters	17
FAJANS, L., Experiments on tannin	256	HIRSCHSOHN, E., The detection of oil and gurgun balsam in copaiba	252
FARSTEINER, K., A method of converting the cuprous oxide obtained in gravimetric estimation of sugar into cupric oxide	258	HORNE, W. D., The rapid and accurate analysis of bone-black	69
FERNANDEZ-KRUG & HAMPE, A new method of estimating milk fat	20	HUGHES, J., Feeding experiments with Indian peas containing <i>Lathyrus sativus</i>	169
FILSINGER, F., On the examination of linseed-oil, raw and boiled	137	HUGOUNENG, L., Estimation of potassium sulphate in wine	184
FREYER, F., Estimation of formic acid	208	" Note on the adulteration of commercial peptones	94
FRIEDHEIM & MICHAELIS, Separation of arsenic from other elements by means of methyl alcohol and hydrochloric acid	235	HUMBERT, M., Chemical process for recognising horseflesh	95
FRESENIUS, R., On the detection of small quantities of metals of the second group in precipitated sulphur	45	J.	
		JANNASCH, P., Lead carbonate as an agent for opening up silicates	191
		JAY & DUPANQUIR, Estimation of boric acid	223
		JEAN, P. F., The determination of free sulphuric acid in leather	62
		JEDERMANN, R., On the detection of geranium-oil in rose-oil	48
		JONES, H. C., On the determination of formic acid by titration with potassium permanganate	207

K.	PAGE
KEBLER, L. F., Acidimetric estimation of vegetable alkaloids	278
Kenrick, E. B., Note on the detection of cotton-seed-oil in lard	136
KILGORE, B. W., On the estimation of phosphoric acid by titration of the molybdate precipitate	17
KILLING, C., Viscometrical examination of butter	66, 94
KIPPENBERGER, C., On the isolation, quantitative separation, and chemical characteristics of alkaloids and glucosidal bodies in forensic cases	201
KITTO, B., Note on a sample of supposed lard	32
KJELDAHL, J., On the action of alkaline copper solutions on sugars	227
KONINK, L. L. DE, The action of magnesia mixture on glass	138
" Volumetric analysis of chloroplatinates, the determination of potassium, ammonium, nitrogen, and platinum	140
KRAAL, H., Preservation of starch solution	23
" Detection of nitrites in presence of iron salts	24
KREIS, H., Modification of Renard's process for the detection of arachis-oil	136
KRUG, W. H., The determination of tannin by metallic oxides	283
KRÜGER, M., The determination of nitrogen in nitrates, nitro-compounds, and nitroso-compounds	45
L.	
LANGE, G., Quantitative estimation of cellulose	283
LEEDS, A. R., Acetic acid in vinegar	224
LEGLER, L., The estimation of zinc in desiccated apples	251
LESOUR, The detection of watered milk by the examination of the milk serum	200
LIDOW, A. P., The influence of temperature on the elaidin reaction	178
LINTNER & KRÜGER, On the employment of phenylhydrazine for the quantitative estimation of dextrose, lævulose, and saccharose	167
LINTON, L. A., On the technical analysis of asphaltum	41
LIVERMEGE, J. F., The composition of milk and the conditions affecting it as shown by Dr. Bell's analyses	7
LUNGE, G., On the use of litmus and methyl-orange as indicators in volumetric analysis	65
M.	
MANCUSO-LIMA & SGARLATA, Determination of glycerol in wines, and indirect estimation of mannitol	183
MANSFIELD, M., Lard	222
MARIANI & TASELLI, Contribution to the study of the ash of cheese	168
MARTELLI, D., Simple method of detecting polvrette in ground pepper	181

PAGE	
61	MAYBURY, C. F., The determination of sulphur in volatile organic compounds
93	McILHINNEY, The analysis of varnishes
235	MIGNOT, A., Volumetric estimation of arsenic in iron and steel
22	MOISSAN, H., On the estimation of boron
223	MOLHANT, M., The detection and estimation of glycerin in beer
236	MYLIUS & FROMM, Preparation of pure zinc
N.	
45	NEGRI & FABRIS, Oils
20	NEUBAUER, H., Method of obtaining pure white magnesium pyrophosphate in the citrate method
252	NIEBEL, W., On the chemical recognition of horseflesh
286	NISSENSON & NEUMANN, The analysis of lead, metal and ores
185	NIVIERE & HUBERT, Detection of fluorine in wine
O.	
298	OETTEL, F., An improved burette
232	OSBORNE, T. B., The chemical nature of diastase
226	OST, H., The determination of starch
258	" The rotatory power of maltose
259	" Estimation of sugar by means of potassium copper carbonate solution
P.	
141	PALADINO, P., On a new alkaloid in coffee
23	PANAOTOVIE, V., The purification of salt-petre from perchlorate
251	PARTHEIL, A., The estimation of glycerin in wine and beer
288	PEARMAN & MOOR, The composition and analysis of condensed milk
174	" Note on adulterated sandal-wood-oil
176	" Note on a sample of adulterated coffee
134	PEARMAN, T. H., The testing of oils by the oleo-refractometer
63	PECKHAM, S. F., The asphalt question
95	PEMBERTON, H., junr., The determination of phosphoric acid
280	PESKA, Z., The volumetric estimation of sugar by means of ammoniacal copper solution
111	PLIMPTON & CHORLEY, The use of barium thio-sulphate in standardizing iodine solution
277	POLENSKI, E., Examination of butter
188	POSETTO, G., The composition of some vegetable colouring matters for use in confectionery
198	PRINSEN, H. C., Ang-Khak, a Chinese dye used for the coloration of articles of food
262	PRIZOZNIK, E., The influence of the metals of the platinum group on the estimation of gold by parting
R.	
47	RAIKOW, P. N., Detection and quantitative estimation of chlorine in the presence of iodine
1	RICHMOND, H. D., Note on Recknagel's phenomenon

	PAGE
RICHMOND, H. D., The composition and analysis of milk and milk products ...	54
„ The relation between specific gravity, fat, and solids-not-fat in milk ...	57
„ Maumené's test for oils ...	58
„ Duclaux's method for the estimation of volatile fatty acids, the laws governing volatility deduced therefrom, and their application to analysis, especially to that of butter ...	193, 217
RICHMOND & BOSELEY, Note on the detection of formalin ...	154
RIDEAL, S., On formalin as a milk preservative ...	157
RIKOWA, P. N., Separation of copper from cadmium, zinc, and nickel ...	25
ROLFFS, J., Test for distinguishing between butter and margarine ...	221
ROODE, R. DE, The addition of calcium chloride to a solution of a fertilizer in the determination of potassium ...	66
S.	
SAMUELSON, J., An abnormal butter ...	221
SANI, G., The estimation of available phosphoric acid in Thomas slag ...	189
SCHAFER, W., The preparation of litmus solution ...	37
SCHAFER, F., The detection of Martins yellow in macaroni, etc. ...	225
SCHIFF & TARUGI, Thioacetic acid as a substitute for hydrogen sulphide in analysis ...	62
SCHNEIDER, J., A new method of estimating indigotin ...	211
SCHNEIDER, L., A new method of estimating carbon in steel ...	65
SCHUTTEN, M. C., The estimation of iodine in organic substances of the fatty series ...	234
SCHWEITZER & LUNGWITZ, The detection of water in acetone ...	234
SHIMER, P. W., Note on the determination of zinc ...	143
SHIVER, F. S., Standardization of sulphuric acid ...	143
SOSTEGNI, L., Methods of analysis of copper salts ...	189
SPAETH, E., The acetyl numbers of lard, cotton-seed-oil, and tallow ...	252
„ The chemical variations of different kinds of mace ...	200
SPALLANZANI & PIZZI, Some samples of Italian butter ...	199
SPICA, M., The detection of salicylic acid in wine ...	184
SPINNETTE, H., Detection of naphthol ...	211
STAHR, L., Characteristic reaction of citric acid ...	188
STANOJEWITSCH, B., On the estimation of nitrogen in nitro-cellulose ...	21
STIFT & PETZIWAL, Preparation of sugar solutions for the polarimeter ...	232
STONE, G. C., Volumetric determination of zinc; a new indicator for ferrocyanide ...	236
STONE, W. E., A comparison of methods for the determination of starch ...	17
STONE & SCHEUCH, A method for determining calcium oxide in quicklime ...	19
STUTZER, A., A comparative examination of the constitution of different meat extracts ...	182
„ On the analysis of the nitrogenous constituents in meat extracts and commercial peptones ...	246
T.	
TARUGI, N., The action of thiacetic acid on various metallic solutions in the cold ...	213
TENNILLE, G. F., The phospho-molybdic acid test in lard analysis ...	63
THRESH, J. C., The interpretation of the results obtained upon the chemical and bacteriological examination of potable waters ...	80, 97
TIEMANN & KRÜGER, Ionone and irone ...	234
TWITCHELL, E., The separation of solid and liquid fatty acids ...	165
U.	
UFFELMANN & BÖMER, On the chemical composition of Cola nut ...	
V.	
VERDRÜDI, V., Method for the quantitative estimation of ammonia in tobacco in presence of nicotine ...	255
VIGNA, A., The determination of tannin in wine ...	184
W.	
WAGNER, P., The estimation of citrate soluble phosphoric acid in Thomas slag ...	215
WALLER, —, The theory of Hübl's iodine process, and a suggestion for its improvement ...	280
WEIGLE & MERKEL, Action of formalin on food stuffs ...	167
WENDR, N., The viscometrical examination of butter for foreign fats ...	220
WENSE, W., On the estimation of bromine in the presence of chlorine ...	139
WHITE, J., Note on the use of maize as an adulterant of oatmeal ...	30
WHITEHEAD, C., Estimation of tellurium in copper bullion ...	189
WILLIAMS, R., Note on the iodine and bromine absorptions of linseed-oil ...	276
WISLIGENUS & KAUFMANN, Amalgamated aluminium as a neutral reducing agent in the presence of water ...	213
WOLFF, C. H., The use of the Mohr-Westphal balance in milk analysis ...	115
WOLFFENSTEIN, R., The concentration and distillation of hydrogen peroxide ...	38
Y.	
YOUNG, W. C., A comparison of the organic carbon and nitrogen results obtained by Dr. Frankland and the companies' analysts from the waters supplied by the Metropolitan Water Companies ...	159

THE ANALYST.

JANUARY, 1895.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE usual Monthly Meeting of this Society was held on December 5th at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner took the chair.

The minutes of the last meeting were read and confirmed.

H. S. Shorthouse, Pershore Road, Birmingham, was proposed for election as member.

The following gentlemen were duly elected: as member, Raymond Ross, Fort Royal, Worcester; as associates, C. A. Mitchell, M.A., and J. Lewin.

On the proposal of Mr. Allen, seconded by Mr. Smetham, Mr. B. Kitto and Dr. F. L. Teed were appointed auditors.

Mr. Richmond then read the following paper:

NOTE ON RECKNAGEL'S PHENOMENON.

By H. DROOP RICHMOND.

RECKNAGEL first observed that the specific gravity of milk, when taken as soon as possible after milking, was lower than observations on the same milk at later periods. He attributed this to a swelling of the casein, though it should be more properly a contraction of the casein.

Vieth (*ANALYST*, xiv., 69) made a series of experiments, and found a rise in twenty-four hours varying from 0.001 to 0.002, and averaging 0.0013, and confirmed Recknagel's statement completely.

Bourcart (*Bull. Soc. Chim.* [3], i., 24) has also made the same observation.

Pappel and I (*Journ. Chem. Soc.*, lvii., 754) have observed in the milk of the gamoose an average rise of 0.0007 in twenty-four hours, and concluded that the rise was not so great in the milk of the gamoose as in that of the cow. The maximum rise observed in twenty-four hours was 0.0009, and the minimum 0.0003. In one sample examined later than the others, I found a decrease in specific gravity, the figures being 1.0351 after two hours, and 1.0345 after twenty-four hours. The duplicate determinations with different pynometers only differed by 0.00002, and I do not think that experimental error was the cause of the difference.

The American Association of Official Agricultural Analysts proposed to touch upon this subject for this year, but their report has not yet reached me. In private communications both Dr. Babcock and Dr. Farrington have informed me that they have either observed no rise or a very slight one.

It appeared to me desirable to further examine the question, and to see if the phenomenon were general, or, if not, under what circumstances it took place, and to determine to what change in the constituents it was due.

The following reasons are possible :

- (i.) When fresh milk is drawn from the udder, minute air-bubbles form, and are suspended in the milk for a long period.
- (ii.) Milk-sugar in the udder may be formed as anhydrous sugar, and the rise may be due to a hydration.
- (iii.) A change in the albuminoid, analogous to that of fibrin, may occur ; in this case it would probably be enzymic.
- (iv.) The fat globules may contract very slowly as the milk cools, owing to their being surrounded by a layer of badly conducting material.
- (v.) A chemical change, such as that between aldehyde and water (cf. Perkin, *Journ. Chem. Soc.*, li., p. 817), may take place.

The reasons may be examined as follows :

- (i.) This reason is not very likely, as microscopic examination with a high power fails to show air-bubbles after about one hour after milking ; froth produced by artificial means disappears in about one hour or less.
- (ii.) This is also most improbable, as milk-sugar solutions do not change in density during chemical change indicated by a rapid variation in specific rotation ; nor does the milk-sugar, estimated by the polariscope, differ when estimated soon after milking, and after the lapse of some hours, though a rise in the specific gravity is taking place.
- (iii.) Salicylic acid has an inhibitive action on many enzymes ; preliminary experiments have given indications of the rise being hindered by salicylic acid.
- (iv.) Is also improbable, as in this case milks rich in fat should show more rise than poor milks ; experiment does not bear this out.
- (v.) Chemical change should follow Harcourt and Esson's laws. Data are not sufficiently numerous to ascertain whether the rise in specific gravity does so, but there are indications that, though in fair agreement, the rise is not wholly in accordance with them.

O'Sullivan and Tompson (*Journ. Chem. Soc.*, lvii., 865) have shown that an enzyme, invertase, also follows to a great extent Harcourt and Esson's laws, and that therefore there is no sharp distinction between chemical and enzymic action.

It seems probable, therefore, that Recknagel's explanation that a change in the casein occurs is correct, and that this is due to an enzyme (possibly the enzyme which causes the change within the udder) ; it would appear from this that milk is still a living tissue after secretion. It is probable, then, that fore-milk (*i.e.*, milk that has remained ready formed for some time within the udder) will show much less rise than the later portions (which are drawn away as secreted). Preliminary experiments have shown indications of this being the case.

I have shown that for two successive years practically the same differences have occurred between the fat calculated and that estimated at different periods of the year (*ANALYST*, xix., 82) ; though I hesitated to place too much reliance

on the apparent concordance, it is interesting to bear this in mind when studying the periodical variations of the Recknagel phenomenon. There is some evidence already that the rise in specific gravity takes place to a much greater extent in the autumn and winter than in the spring and summer, and that the year can be divided into periods corresponding to those indicated by the agreement of the calculated fat. Much systematic work will, however, be necessary to establish the views that I have indicated.

I reserve quantitative details for a later communication, but I may say that while, in some cases, the rise has been as great as that found by Recknagel and Vieth, in others it has been absolutely nil.

DISCUSSION.

Mr. Alfred Smetham said that in a series of milk analyses which he had conducted in connection with some experiments in Cheshire cheese-making, there had occurred considerable differences between the percentages of fat as calculated from the specific gravities (which were taken at the farm where the cheese was being made) and the amounts arrived at from determinations made in his laboratory by the Adam's process. These differences were so marked as to cause him to distrust the hydrometer with which, in the first instance, the specific gravities were taken; but the instrument, when tested, was found to be quite correct; and, moreover, the discrepancies still remained when a balance had been substituted for the hydrometer. A rise in the specific gravities of the samples seemed to have taken place during the time which elapsed between the measurements at the farm (which circumstances rendered it necessary to make within half an hour of milking) and the analyses made in the laboratory. After careful observation and consideration, he came to the conclusion that this must be due to the gas, as of the milk and the air bubbles mechanically carried into the milk during the operation of milking, which would disperse after some lapse of time. The experiments extended over several months of two successive years, but were not continuous, taking place in the late summer and autumn only of each year.

Dr. Sykes said he thought that enzymic action might possibly account for the phenomenon under consideration. It was now a well-ascertained fact that the peptonization of proteids was a process of hydrolysis, and this would no doubt be accompanied by a rise in the specific gravity of the solution of the proteid, just as a solution of cane-sugar, owing to fixation of water by the sugar molecule, becomes specifically heavier under the action of the enzyme invertase.

In the absence of the respective authors, Dr. Dyer read a paper entitled "Note on Woody Fibre Determination," by A. P. Aitken, D.Sc.; also "Note on a Sample of Red Water," by W. F. Lowe; and the three following papers:

THE ANALYSIS OF SODIUM PEROXIDE.

By L. ARCHBUTT, F.I.C.

SHORTLY after sodium peroxide became an article of commerce, I had occasion to make a full analysis of a sample of it, which led to some observations of interest. As I am not aware that a really accurate, rapid method for the assay of this article has been published, I venture to bring the results of my experiments before the Society.

The sample of the peroxide which I examined was taken from a full 14-lb. canister fresh from the makers' works. A portion of it was transferred to a stoppered tube, and all the weighings were made by taking some from the tube, which was immediately re-stoppered and the loss of weight ascertained.

Estimation of Soda (Na_2O).

About 0.8 gramme was accurately weighed, dissolved in water, boiled to decompose the peroxide, cooled, tinted with methyl orange, and titrated with normal sulphuric acid. A few drops more of the acid were then added, the solution evaporated to dryness in a tared platinum dish, and the residue weighed after ignition. From the weight obtained there was deducted the weight of the alumina and iron oxide found by another experiment, and the difference was calculated to Na_2O . Two experiments gave :

Na_2O by titration, 78.60 and 78.65. Mean	78.63
Na_2O weighed as Na_2SO_4 , 78.72 and 78.69. Mean	78.70
General Mean	78.66

Estimation of Alumina and Iron Oxide.

About 2 grammes were taken, dissolved in water, acidified with hydrochloric acid, and precipitated by ammonia in slight excess :

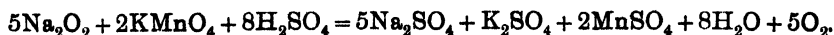
Weight of ignited ppt.	Per Cent. 0.49
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Estimation of Oxygen existing as Peroxide.

First Method tried.—From 0.2 to 0.25 gramme of the sample was weighed into a small dry tube closed at one end, which was then placed in the flask attached to a Lunge nitrometer. The flask contained a small volume of potassium permanganate solution acidified with sulphuric acid, and after the whole had acquired the temperature of the room, the mercury was levelled, and then the peroxide and the permanganate solution were brought into contact.

Weight taken in grammes.	Oxygen obtained in c.c., reduced to 0° and 760 mm. (dry).	Oxygen obtained in grammes.	Oxygen obtained per cent., assuming half came from the KMnO_4 .
0.1974	29.78	0.0426	10.79
0.2434	37.28	0.0533	10.95
0.2457	38.56	0.0551	11.22
0.2447	38.47	0.0550	11.24

Evidently the decomposition was only partially effected in accordance with the equation :



Second Method tried.—About 0.1 gramme was dissolved in cold water previously mixed with some dilute sulphuric acid, and the solution was titrated with decinormal permanganate. Three experiments gave 18.4, 18.3, and 17.8 per cent. of oxygen.

There was a slight loss of oxygen at the moment of contact between the peroxide and the acidified water. A larger quantity, 1.0019 grammes, was dissolved in acidified water, diluted to 1 litre, and 100 c.c. titrated. Two experiments gave only 17.45 per cent. of oxygen.

Third Method tried.—An attempt to decompose the peroxide in the nitrometer with water alone was not successful, decomposition being much too slow and incomplete. But a trace of cobalt nitrate previously added to the water was found to be all that was required to effect rapid and complete decomposition. The peroxide was weighed into the small tube, and into the flask were placed about 5 c.c. of water and one small drop of cobalt nitrate solution. Three experiments were made, and gave the following results:

Weight taken in grammes.	Oxygen obtained in c.c., reduced to 0° and 760 mm. (dry).	Oxygen obtained in grammes.	Oxygen obtained per cent.
0.2499	32.40	0.0463	18.54
0.2472	31.91	0.0456	18.46
0.2537	32.94	0.0471	18.57

The mean of these results—viz., 18.52 per cent.—was accepted. They show that decomposition by water in the nitrometer flask in the presence of a milligramme or two of cobalt sesquioxide is the most accurate, as well as the simplest, method for the assay of sodium peroxide. It would be more accurate to add precipitated cobalt sesquioxide than cobalt nitrate, as obviously a trace of oxygen is used up in oxidizing the cobaltous oxide. My result may, therefore, be a trifle too low; but, taking it as practically correct, the composition of the sample works out as follows:

Constituents found.				Calculated composition.			
			Per cent.				Per cent.
Na ₂ O	78.66	Na ₂ O ₂	90.41
O	18.52	NaOH	8.73
Al ₂ O ₃ and Fe ₂ O ₃	0.49	Al ₂ O ₃ and Fe ₂ O ₃	0.49
							99.63

This analysis does not take into account a very little carbonate, a trace of chloride, a minute trace of sulphate, and a few particles of unoxidized sodium which the sample contained. No other impurity was detected.

DISCUSSION.

Dr. Dupré said that the analysis of sodium peroxide was a very unsatisfactory operation to perform. All the processes he had tried were almost impossible to conduct without loss of oxygen. He had not, however, tried the cobalt process, but thought it would be a sensible improvement.

NOTE ON GLADDING'S PROCESS FOR THE DETERMINATION OF RESIN IN SOAP.

By L. ARCHBUTT, F.I.C.

LEWKOWITSCH's destructive criticism of all the methods which have been proposed for the determination of resin in soaps* leaves Gladding's process in this position: that, in the absence of a really reliable process, it will continue to be used on account of its simplicity, especially in cases where the soap has been made from a known fat. I wish to direct attention to a source of error which is one cause of erroneous results.

In his original paper, "On the Quantitative Separation of Resin from Fats,"† Gladding directs that 0·5 gramme of the mixed fatty and resin acids is to be dissolved in 20 c.c. of 95 per cent. alcohol. The solution, mixed with one drop of phenolphthalein, is to be treated with a saturated alcoholic solution of caustic potash, added drop by drop until the deep-red colour characteristic of alkalinity is obtained, after which "one or two additional drops of the potash solution are added, and the flask is placed on the water-oven and kept at the temperature of boiling alcohol for ten minutes to ensure the saponification of the last traces of fat." In the analysis of soap, "one gramme of the soap in thin shavings is dissolved in the alcohol, and treated in every way as above. Particular care is paid to the saponification, as above directed, in order to make certain that no trace of unsaponified fat is left."

In my laboratory the above directions have always been carried out, but I have long been sceptical as to the value of the last part of the process, by which the absence of unsaponified fat is supposed to be ensured. On not a few occasions the resin acids extracted from soaps have been more or less soft, instead of hard and brittle. But my observations have not gone beyond this, until a few days ago, when two samples of palm soap were being tested side by side; from one the resin obtained was quite hard and brittle, and from the other soft. As the soft resin was several units per cent. higher than the hard resin, and the question under investigation related to the relative amounts of resin in the two soaps, it became necessary to make further experiments. So 10 grammes of the soap yielding the soft resin were dissolved in alcohol, and boiled down in a basin with 1 c.c. of a 50 per cent. caustic soda solution. The soap, thus thoroughly freed from unsaponified oil, was dissolved in hot water, the solution well boiled, and decomposed with dilute sulphuric acid. A suitable quantity of the washed and dried fatty and resin acids was weighed, dissolved in alcohol, neutralized with potash, and the resin acids separated as usual. They were now quite hard and brittle. The results numerically were as follow:

				Resin Acids.
First experiment	25·18 per cent, soft.
Second	„	21·07 „ hard and brittle.

The crude fatty and resin acids obtained from a soap in the ordinary course of analysis would contain the unsaponified oil, and would be no better to work upon than the soap itself. The soap must, therefore, either be boiled with a sufficient

* *Soc. of Chem. Ind.*, 1893, pp. 503-508.† *Chem. News*, vol. xlv., p. 160.

excess of alkali to saponify undecomposed fat, or else the aqueous solution of the soap must be shaken with ether to remove unsaponified oil, and the acids liberated from the purified soap solution used for the resin estimation. The latter method would be preferable, as it would remove any cholesterol or other unsaponifiable matter as well as the unsaponified oil.

DISCUSSION.

Mr. Allen said that he had found it a great improvement to precede Gladding's process by a preliminary treatment of the soda soaps of the fatty and resin acids with ether-alcohol, as directed by Barfoed. This left the greater part of the fatty acids insoluble, and it became much easier to effect a separation of the resin acids from the residual fatty acids by Gladding's silver process. But of late he, Mr. Allen, had rather preferred Twitchell's process to Gladding's, even with the preliminary Barfoed treatment. This method consisted in passing hydrochloric acid gas through a solution of the fatty and resin acids in absolute alcohol, whereby the former suffered conversion into compound ethers or esters, while the resin acids were unaffected. On diluting the product with water, and adding petroleum spirit, the resin acids and esters were readily separated from the aqueous liquid containing the hydrochloric acid, and the amount of the resin acids ascertained approximately by titrating the washed layer with standard alkali. The results so obtained could be checked by separating the lower layer, acidulating it, and weighing the liberated resin acids. There was no doubt that the exact determination of resin in soap, etc., was a very difficult problem, which could not be regarded as completely solved, and any approach to greater simplicity or accuracy would be welcomed by those accustomed to make such determinations.

Mr. Smetham said his experience of Gladding's method coincided with that of Mr. Allen. Where a large number of determinations had to be made, and something was known about the constituents of the soaps, the easiest and best plan was to compare the specific gravities of the resin and fatty acids with the specific gravities of standards made up of known quantities of resin and fatty acids from various sources, the specific gravities being taken at 100° C. In this way the percentages of resin and fatty acids could be determined with sufficient accuracy for manufacturing and commercial purposes. If the sources of the fatty acids were not known, determinations of the saponification equivalent and of the iodine absorption would, as a rule, give sufficient information to enable such cases to be dealt with.

THE COMPOSITION OF MILK, AND THE CONDITIONS AFFECTING IT, AS SHOWN BY BELL'S ANALYSES.

By J. F. LIVERSEEGE, F.I.C.

DR. BELL's analyses of milk, dated August 15, 1893, have several times been quoted in police-court proceedings, and it seemed worth while to examine them in detail, to ascertain how far they can be quoted in opposition to this society's standard, particularly as the report accompanying them is so meagre.

The average composition is shown by the following table :

Averages.	Sp. Gr.	Solids per Cent. (Bell).			Calculated.	
		Not-Fat.	Fat.	Total.	S.-n.-F.	Fat.
273 Cows	1032.3	8.908	3.991	12.899	8.86	4.03
55 Dairies	1031.9	8.96	4.00	12.96	8.80	4.16
Dr. Vieth	—	—	—	12.90	8.8	4.1

The solids-not-fat and fat have been calculated from Hehner and Richmond's table, so that the results could be compared with Vieth's average of 120,540 samples. It will be seen that these averages are practically identical, though it is somewhat remarkable that in the dairies the calculated fat exceeds the found by .16 per cent., while in the single cows the excess is only .04 per cent.

The range in quality is shown by the following table. I have also calculated all the low figures of the single cows for comparison with the Society's standard.

55 DAIRIES.		273 Cows.	
Total Solids.			
Under 11.5 %	0 Milks = 0 %	7 Milks = 3 %	
11.5 to 11.99	4 " 7	37 " 14	
12.0 " 12.49	6 " 11	69 " 25	
12.5 " 12.99	19 " 35	44 " 16	
13 " 13.99	23 " 42	82 " 30	
14 " 14.99	3 " 5	22 " 8	
15 and over	0 " 0	12 " 4	
	55 100	273 100	

Fat.	Bell.		Bell.	Calculated.
Under 2.75 %	0 Milks = 0 %		6 Milks = 2 %	3 Milks = 1 %
2.76 to 2.99	1 " 2	12 " 5	10 " 4	
3.0 " 3.19	1 " 2	21 " 8	260 " 95	
3.2 " 3.49	8 " 15	37 " 14		
3.5 " 3.99	18 " 32	75 " 27		
4 " 4.99	26 " 47	95 " 34		
5 and over	1 " 2	27 " 10		
	55 100	273 100	273 100	

Solids-not-Fat.	Bell.		Bell.	Calculated.
Under 8 %	0 Milks = 0 %		5 Milks = 2 %	7 Milks = 3 %
8.0 to 8.29	0 " 0	10 " 4	17 " 7	
8.3 " 8.49	2 " 4	31 " 11	40 " 15	
8.5 " 8.69	6 " 11	33 " 12	209 " 75	
8.7 " 8.99	21 " 38	95 " 35		
9.0 " 9.49	25 " 45	71 " 26		
9.5 and over	1 " 2	28 " 10		
	55 100	273 100	273 100	

It will be seen that no dairy gave less than 11·5 per cent. of total solids, the lowest being 11·68 per cent. and 11·80 per cent., while the lowest single cows were 10·33 per cent., 11·19 per cent., 11·21 per cent., and 11·35 per cent., the other three being more than 11·4 per cent.

Among the dairies, the only one below 3 per cent. of fat is 2·89 per cent., which when calculated, gives 2·99 per cent. The two lowest single cows give 2·43 per cent., and calculate to 2·6 per cent. and 2·67 per cent. The seven milks with total solids under 11·5 per cent. have all less than 3 per cent. of fat.

Two of the dairy milks give solids-not-fat under 8·5 per cent., viz., 8·4 per cent. and 8·48 per cent.; but four of the calculated figures are below the standard, viz., 8·28 per cent., 8·33 per cent., 8·35 per cent., and 8·42 per cent. The two lowest single cows are 7·52 per cent. and 7·79 per cent., and the two lowest calculated are 7·52 per cent. and 7·66 per cent.

In all, 74 milks from single cows are below the Society's limits—10 being low in fat only, 61 in solids-not-fat only, and 3 in both; but in 37 cases there is a compensation in the other constituents, so that the total solids are 12 per cent. or over, while 26 more are within 0·2 per cent. of the standard, leaving only 11 milks, or 4 per cent., that are much below the standard.

It has been stated that the coil process gives "an increase of what is called butter-fat of perhaps 0·2 per cent." over the Bell process, but it is worth while noting that sometimes the calculated fat exceeds the figures given by 0·4 per cent.; in other cases the calculated fat is lower—in one case as much as 0·26 per cent.

As in each milk, the age, food, hour of milking, and time from calving are given, as well as the breed of the cows, and the quantity of milk yielded, I tried to find if, from these analyses, any general conclusion as to the effect of these factors on the quantity of the milk could be arrived at. It might be thought that these things do not concern the members of the Society, but as the public analyst may any time be cross-examined as to the effect of food, etc., I do not think any apology is needed.

The following table gives the average composition and quantity of milk yielded by the cows of the various breeds, but it should be noticed that the number of samples in some cases is too small to be of much use.

The milk from the North Devon cows is highest in solids-not-fat and lowest in fat. The Dutch cows give lowest total solids and solids-not-fat, while the Jersey milks are highest both in total solids and in fat. The cross-breeds give the greatest, and the Kerries the least yield of milk:

Breed.	Number of Cows.	Solids-not-Fat.	Fat.	Total Solids.	Average Yield. Quarts.
Sussex	2	8·92	3·39	12·31	7·5
Welsh	2	9·15	4·40	13·55	4·2
Guernsey	4	9·30	5·16	14·46	4·1
Jersey	6	9·22	5·43	14·65	3·7
Kerry	5	8·87	4·67	13·54	2·7
North Devon	6	9·68	3·43	13·11	3·8
Dutch	13	8·65	3·75	12·40	5·3
Ayrshire	15	9·22	4·24	13·46	4·6
Cross-breeds	17	9·01	3·99	13·00	5·6
Shorthorns	203	8·86	3·92	12·78	5·3
General Average ...	273	8·908	3·991	12·899	

As there was such a difference in the various breeds, it was deemed advisable to eliminate this factor and investigate the shorthorns only. It is well known that morning milk is poorer than evening, and it has been suggested that this is due to the difference of time elapsing between milking. To test this, the facts with regard to each cow were copied on a strip of cardboard, and the strips sorted. For solids-not-fat they were divided into three classes—under 8·5¹ per cent., 8·5 per cent. to 9 per cent., and 9 per cent. and over—and each class subdivided into first meal and second meal, and the numbers in each class counted and calculated to percentages. Thus, 23 per cent. of the milks under 8·5 were first meal, and 77 per cent. second meal. The fats were similarly classified, and then both were arranged with regard to hours from last milking.

Figures on the same horizontal line may be compared, but vertically each class adds up to 100 :

	Solids-not-Fat.			Fat.			Average Quantity. Quarts.
	Under 8·5.	8·5 to 8·99.	9·0 & over.	Under 3·5.	3·5 to 4·49.	4·5 & over.	
First meal ...	23	29	29	55	21	5*	
Second meal ...	77	71	71	45	79	95	
Hours between milking :							
8 to 9 hours	30	29	30	11	28	60	4·4
10 „ 11 „	48	41	37	29	50	36	5·1
12 „ 13 „	17	16	16	24	17	2*	6·4
14 „ 16 „	5*	14	17	36	5	2*	5·9

This table shows that while 55 per cent. of the milks low in fat were first meal, only 5 per cent. of those rich in fat belong to this meal. Also that, while 11 per cent. of poor milk were 8 to 9 hours between milking, 60 per cent. of the milks high in fat were in this class, showing that the longer the time between milking, the larger will be the proportion of milks low in fat, a conclusion which is supported by the other figures. If, however, the solids-not-fat be examined, it will be seen that time makes practically no difference, but a longer interval has a slight tendency to decrease the proportion of poor milks.

This shows that for the study of the effect of food on the amount of fat, etc., it is necessary to select either first or second meal cows, or the differences due to the meal would mask the smaller variations ; the second-meal class, being the larger (147 cows), was selected, but all shorthorns were taken for the solids-not-fat :

* Number of cows small.

		Solids-not-Fat.			Fat.		
		Under 8.5.	8.5 to 8.99.	9.0 & over.	Under 3.5.	3.5 to 4.49.	4.5 & over.
Age	2 to 4½ years	11*	34	32	31	33	24
	5 " 6 "	62	36	46	54	39	65
	7 " 8 "	16	24	22	11*	19	11
	9 " 12 "	11	6	0	4*	9	0
Yield	1 to 3½ quarts	25	23	28	19	22	37
	4 " 5 "	45	27	31	39	41	30
	6 " 7 "	17	37	27	23	30	30
	8 " 10 "	13	13	14	19	7	3
Time from Calving	1 to 6 weeks	18	28	30	15*	28	27
	7 " 12 "	37	36	16	38	33	25
	3½ " 6 months	27	23	30	31	28	18
	7 " 9 "	10*	8	16	8*	6	27
	10 months & over	8*	5	8	8*	5*	3*
Percentage of cows fed on							
	Hay	20	48	32	17	55	28
	Mangolds or Swedes ...	22	47	31	16	55	29
	Brewers' or distillers' grains	21	43	36	14	50	36
	Linseed or cotton-seed cake	25	45	30	16	62	22
	Chaff or straw	25	49	26	17	64	19
	Meals (oat, pea, barley, etc.)	18	53	29	19	62	19
	Grass	15	56	29	19	56	25

With regard to age, it may be noted that cows five and six years old give a large proportion of the milks, and that no milks high in fat or solids-not-fat occur in milk from cows over eight years old.

When the quantity of milk is under one gallon, the proportion of milks high in fat is increased, and when two gallons and over is yielded at a milking, there is a small proportion of rich milks. The milks given by cows for periods of less than seven weeks after calving have a small proportion poor both in fat and solids-not-fat, and those seven to nine months after calving are of a better quality, particularly in fat.

With regard to the effects of food, the problem is very complicated, as the majority of cows are fed on from three to six different kinds. The figures given are the percentage of rich, medium, and poor milks yielded by cows fed on each food considered by itself—thus, out of 100 cows fed on hay, 20 gave milk that was low, 48 medium milk, and 32 milk high in solids-not-fat. Some of the cows were fed on grass only, and with regard to part of them it is remarked that the grass was very scarce. The very small variations appear to show that food by itself has not much effect on the composition of the milk if the cows are fairly fed, as these are supposed to be. The large proportion of good milks from cows fed on grains is notable.

* Number of cows small.

The one respect in which the information given about these milks is deficient, is that no date is stated, and as the time of the year exercises a marked effect on the composition of the milk, one feels that, as this could not be allowed for, the influence of season may have introduced some errors into the above results. To summarize what has been said, these analyses show

(1) That, with four very slight exceptions, all the mixed milks are up to the Society's standard.

(2) That only 4 per cent. of the milks from the single cows would show signs of much adulteration, as judged by the Society's standard, if allowance is made for excess of the other constituents.

(3) That analyses by the Bell method may give from 0.40 less to 0.26 per cent. more than the calculated figures.

(4) That the longer the time between milking, the lower will be the fat in milk.

(5) That, with the exception of breed, the other conditions have much less effect on the proportion of poor milks.

ON ROASTED CHICORY.

By E. G. CLAYTON.

(Read at the Meeting, October 29, 1894.)

THE amount of sugar present in roasted chicory is very variously stated; and in most cases, apparently, only the sugar before inversion has been estimated. The following figures, compiled from Allen's "Commercial Organic Analysis" and other sources, represent the quantities of sugar mentioned by different observers; and some other numbers are quoted in the table for comparison:

TABLE I.

		Sugar before inversion (Glucose).	Sugar after inversion (expressed as Sucrose?).	Total Sugar.	Proteids.	Water.	Mineral Matter.
Petermann	a. Coarse grains.	26.12	—	—	6.38	16.28	7.16
	b. Fine powder.	23.76	—	—	6.64	16.96	8.44
Krauth*	...	22.39	21.18	43.57	—	4.30	10.36
Hassall	a. ...	12.2	—	—	—	14.5	4.3
	b. ...	10.4	—	—	—	12.8	6.8
König†	...	15.87	—	—	6.09	12.16	6.12
Graham, Stenhouse, and Campbell	a. ...	11.98	—	—	—	—	—
	b. ...	15.96	—	—	—	—	—
	c. ...	17.98	—	—	—	—	—
	d. ...	9.86	—	—	—	—	—

* Krauth's figures (except for moisture) originally referred to the substance dried at 100°, but for the above table they have been re-calculated.

† The number obtained by König, given in column 1, is merely stated to be "sugar," and almost certainly refers to glucose only.

Had the proportions of sugar after inversion been also determined, it is probable that very considerable numbers would have replaced the blanks in columns 2 and 3; and in the light of some results about to be summarized, certain of the figures in column 1 are so high as to suggest the possibility that some of the samples contained added sugar. This is further indicated by the low proteids (column 4).

Specimens of whole chicory root and (for comparison) dandelion root were carefully roasted and ground in the writer's laboratory, and extracted with boiling water. The solutions were decolorized by treatment with lead acetate, filtered after the addition of sodium carbonate, and titrated, before and after inversion, with Fehling's solution. A sample of commercially-roasted and ground chicory, purchased as pure, was similarly treated, with the exception that hot alcohol was in this case used, in place of water, for the extraction. The proportions of mineral matter were determined in the two laboratory samples, and are here stated, together with the other results:

TABLE II.

	Matter reducing Fehling's solution before inversion (expressed as Glucose).	Matter reducing Fehling's solution after inversion (expressed as Glucose).	Matter reducing Fehling's solution after inversion (expressed as Sucrose).	Total (i.e., the figures in columns 1 and 3), expressed as Glucose + Sucrose.	Mineral matter.
Chicory root (roasted in laboratory)...	5.66	14.34	13.62	19.28	5.18
Dandelion root (roasted in laboratory)...	5.66	20.43	19.41	25.07	4.05
Chicory root (commercially roasted and ground) ...	7.19	21.58	20.50	27.69	

It will be seen that the numbers for sugar before inversion (column 1) are considerably lower than the corresponding figures in the analyses previously cited.

Some samples of roasted chicory, undoubtedly containing added saccharine matter, lately came into the writer's hands, and yielded the following results on partial analysis:

TABLE III.

	Matter reduc- ing Fehling's solution before inversion (ex- pressed as Glucose).	Matter reduc- ing Fehling's solution after inversion (ex- pressed as Glucose).	Matter reduc- ing Fehling's solution after inversion (ex- pressed as Sucrose).	Total (i.e., the figures in cols. 1 and 3), ex- pressed as Glucose + Sucrose.	Proteids.	Water.	Mineral matter.
Sample 1 (coarse grains or nibs) ...	16.66	46.50	44.17	60.83	—	—	—
Sample 2 (pow- der) ...	12.00	62.50	59.37	71.87	—	—	—
Sample 3 (coarse grains or nibs) ...	13.53	33.75	32.06	45.59	8.19	—	—
Sample 4 (pow- der) ...	9.24	23.61	22.43	31.67	9.31	1.71	6.25

It is obvious that these samples contained large proportions of foreign saccharine matter, if the number in Table II., representing the amount of sugar expressed as sucrose in the laboratory-roasted chicory root, be provisionally taken as a basis of calculation. The unusually small percentage of water found in the fourth sample is noteworthy; and it will be observed also that, in spite of the presence of considerable amounts of sugar, the quantities of proteids in samples 3 and 4 are larger than the proportions quoted in Table I.—already alluded to as being markedly low.

The intensity of the colour of the infusions led to some experiments as to the most suitable methods of extraction and decolorization; a series of estimations of glucose and invert sugar being made, after various means of removing the colour had been employed. An opportunity, of which the writer gladly avails himself, here arises of acknowledging the very careful assistance in these sugar determinations willingly rendered him by Mr. Sidney B. Wright.

The results obtained are collated in Table IV.

TABLE IV.

	Infusion decolorized by	Sample 1 (Grains).		Sample 2 (Powder).		Sample 3 (Grains).		Sample 4 (Powder).	
		Sugar, expressed as Glucose.		Sugar, expressed as Glucose.		Sugar, expressed as Glucose.		Sugar, expressed as Glucose.	
		Before inver- sion.	After inver- sion.	Before inver- sion.	After inver- sion.	Before inver- sion.	After inver- sion.	Before inver- sion.	After inver- sion.
Extracted with water.	Lead acetate only	16.66	46.50	12.00	62.50	—	—	—	—
	Lead acetate only	15.79	44.21	12.50	62.50	13.53	33.75	9.24	23.61
	Lead acetate, fol- lowed by filtra- tion through animal charcoal	15.00	43.07	—	—	—	—	—	—
	Milk of lime only	7.14	47.40	—	—	—	—	—	—
	Lead acetate and milk of lime to- gether ...	0	14.28	—	—	—	—	—	—
	Lead acetate and milk of lime, followed by fil- tration through animal charcoal	—	—	—	—	2.11	24.28	0	18.75
Extracted with alcohol.									

From the above results of duplicate determinations in samples 1 and 2, it is clear that the difference, caused by using alcohol in place of water for the extraction of the sugar, is inconsiderable. As for the several processes used to decolorize the solutions, the results in the first of the four main columns of the table (all of which results were yielded by the same sample, No. 1, and are in this respect strictly comparable), show that filtration through animal charcoal, though removing some

colouring matter which lead acetate had failed to precipitate, effected this at the expense of a small but perceptible reduction in the percentages of sugar. Milk of lime, alone, yielded a pale-tinted solution, but removed nearly one-half of the "sugar before inversion." The addition of lead acetate, followed immediately by milk of lime, resulted in a nearly colourless solution; but practically all the glucose, and more than two-thirds of the "sugar after inversion," had vanished. Confirmation of these results appeared in the numbers yielded by samples 3 and 4 (columns 3 and 4): after the use of lead acetate and milk of lime, followed by filtration through animal charcoal, far lower results were obtained than when lead acetate only had been employed. In short, the use of milk of lime, alone or in conjunction with lead acetate, is quite inadmissible, as, perhaps, might have been anticipated; and the process of extraction with hot water, followed by simple decolorization with lead acetate, appears most suitable in the case of chicory. Filtration through animal charcoal may, in addition, be necessary in many instances, and the influence on the final results will not be very serious.

ADDENDUM.

Since the above was written I have read the evidence on chicory given before the Food Products Committee, and I venture to quote a portion of it here.

After Mr. Bannister's remark, that "there is a greater extractive got from chicory now than there used to be ten or fifteen years ago," questions 866 and 867 followed:

"866. Is that agreed among chemists, or is it owing to your improved methods of analysis?—It is on account of the *difference of growth*, I believe, and not to improved methods of analysis.

"867. That the quality of chicory has improved?—It contains more extractive matter, and is *richer in sugar*." [Italics are mine.]

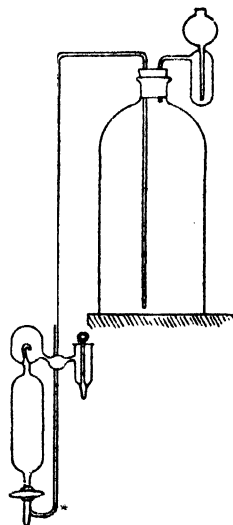
If roasted chicory be referred to (and this is presumably the case), may not the explanation of the greater richness in sugar and extractive matter be found, *not* in any "difference of growth," but in a frequent practice of such extraneous addition as that described in the above paper?

APPARATUS FOR PRESERVING AND DELIVERING STANDARD SOLUTIONS.

BY J. C. CHORLEY.

(Read at the Meeting, November 7, 1894.)

THIS apparatus will be found useful for preserving and delivering known volumes of such solutions as alcoholic potash, which are liable to contamination by exposure to air. The wash-bottle inserted in the large store-bottle is filled with a solution of caustic potash, and so also are the bulbs connected with the top of the pipette. By means of the two-way cock, this can be filled to overflowing, and a known quantity of the solution can be delivered by running off to a mark on the narrow part of the pipette.



only be explained on the supposition that the honey has been mixed by the bees with honey-dew. An increase in the normal amount of mineral constituents and nitrogenous matter may also point to the same conclusion.

2. Direct dialysis of the honey by Hänle's method* is not advisable, since even after 40 to 50 hours sugar is left on the membrane; and on the other hand dextrin has passed through. The difficulty is obviated by dialysing the fermentation products instead of the fresh honey.

3. The dextrans of honey-dew probably undergo some change in the honey-bag of the bee, so that their power of dialysis is increased. C. A. M.

Zinc-bearing Spring Waters. W. F. Hillebrand. (*Bulletin U.S. Geological Survey*, No. 113. *Report of Chemical Work in 1891-92 and 1892-93*).—Analyses are given of two spring waters from Missouri, both containing zinc sulphate as a natural constituent. One contained 120.5 parts of Zn and 61.1 of Ca per million. The other 132.4 of Zn and 63.1 of Ca. A peculiarity in the waters was that, although some of the Ca existed as carbonate, this being shown by the excess of bases over Cl and SO₃, yet *all* the CO₂ was expelled by simply boiling the water. The amount of CO₂ obtained was 120.5 per million in the first water, and 110.0 per million in the second, and no more could be obtained by acidifying the waters. The explanation is that the CaCO₃ at a temperature near the boiling-point of the water reacts with the ZnSO₄, forming CaSO₄, zinc hydrate or a very basic sulphate, and CO₂. A determination of the residue, by simply evaporating the water to dryness would therefore involve a serious error.

C. A. M.

On the Estimation of Phosphoric Acid by titration of the Molybdic Precipitate. B. W. Kilgore. (*Jour. Amer. Chem. Soc.*, 1894, pp. 765-772.)—A summary of different processes is given. The author obtained good results in general by Pemberton's (*ANALYST* xix., p. 135) method, though high ones were not uncommon. The variations were found to be due to deposition of molybdic acid from the molybdate solution. By precipitation at a temperature of from 55° to 60° instead of at 100°, and by the use of the official molybdate solution of the Association of Official Agricultural Chemists instead of the more dilute solution used by Pemberton, this error may be avoided. The results tabulated agree well with those obtained by gravimetric determination. The official molybdate solution is prepared by dissolving 100 grammes of molybdic acid in 417 c.c. of ammonia (sp. gr. 0.96) and pouring this into 1250 c.c. of HNO₃ (sp. gr. 1.20). C. A. M.

A Comparison of Methods for the Determination of Starch. W. E. Stone. (*Jour. Amer. Chem. Soc.*, 1894, xvi., No. 11, pp. 726-733.)—A comparative examination of different methods of starch analysis proves that, while the ordinary methods give excellent results with pure starch, the results obtained with substances

* *Zeit. für Anal. Chem.*, 33-99.

containing other carbohydrates are discordant. This is due to the pentosans or gums acting towards some reagents precisely like starch.

The table gives the results obtained by the following five methods.

1. *Sachsse's Method*.—From 3 to 5 grammes of the material are heated in the water-bath for three hours with 200 c.c. of water and 10 c.c. of concentrated HCl. The glucose formed is estimated by Fehling's solution. According to Märker, this method gives too low results, owing to destruction of glucose by the long heating with the acid.

2. *Guichard's Method*.—Three grammes of the material are heated in the water-bath for one hour with 100 c.c. 10 per cent. nitric acid, made up to 200 c.c. and polarized. The readings are calculated to glucose.

3. Three grammes of the material are heated in the water-bath with 100 c.c. of saturated solution of oxalic acid for an hour, in order to bring the starch into solution. The liquid is cooled, made up to 200 c.c. with 10 per cent. nitric acid, and heated in the water-bath for another hour. The solution is then polarized.

4. *Baudry's Method*.—A normal weight, 5.376, is adopted for the Laurent polarimeter. This weight, or some multiple of it, is placed in a 200 c.c. flask, with 100 c.c. of water, 0.4 to 0.5 of salicylic acid added, and heated in the water-bath for half an hour. The contents of the flask are made up to within 20 c.c. of the mark with hot water, rapidly cooled, a few drops of ammonia added to clarify, filled up to the mark, filtered, and polarized in a 400 mm. tube.

5. *Asboth's Method*.—See ANALYST, vol. xii., p. 138.

Materials.	Inversion by HCl.	Inversion by HNO ₃ .	Inversion by oxalic acid and HNO ₃ .	Solution by salicylic acid.	Precipita- tion by Ba(OH) ₂ .
1. Potato starch	85.75	85.5	85.75	85.47	85.58
2. Dried potato	70.92	69.79	68.53	—	64.25
3. Wheat flour	77.69	70.65	65.29	69.38	59.76
4. Corn meal	73.24	66.81	70.55	—	62.11
5. Wheat bran	65.86	40.25	38.68	—	70.77
6. Hay...	3.48	19.10	19.10	—	66.47
7. Wheat middlings	30.00	63.09	60.24	—	60.44
8. Cotton-seed meal	4.15	—*	—*	—	54.65
9. Mixture of starch, sugar, and dextrin	9.58	21.00	24.08	18.8	33.99

The potato-starch, but for moisture and cellulose, was "pure." Each mode gave credit for the entire amount of real starch present.

The following experiment proves that the pentosans behave like starch. Xylan, free from other carbohydrate, was obtained from wheat straw. One gramme was heated for thirty minutes with 50 c.c. of water and 4 c.c. of strong HCl. The neutralized solution was made up to 100 c.c. Of this, 6.7 c.c. were required to reduce 10 c.c. of Fehling's solution, a quantity equivalent to 67.16 per cent. of starch. The other methods gave similar results, with the exception of the salicylic acid method, which was found not to be applicable.

* These solutions were laevo-rotary.

The author, therefore, concludes that no method based on the foregoing principles is suitable for accurate work on grains or forage material. He suggests the use of some method by which the starch may be brought into solution without other carbohydrates, *e.g.*, by means of diastase or malt infusion. The weighed material, after being made into a paste with boiling water, is cooled to 65°, a small quantity of malt infusion added, and the temperature maintained at from 60° to 65° for half an hour. After the starch reaction has disappeared, the solution is filtered, the residue well washed, and the filtrate, after warming with a few c.c. of acid and then neutralizing, is made up to definite volume and titrated. The preliminary experiments cited indicate that the pentosans and similar bodies are not affected by diastase.

C. A. M.

A Method for Determining Calcium Oxide in Quicklime. W. E. Stone and F. C. Scheuch. (*Jour. Amer. Chem. Soc.*, 1894, xvi., No. 11, pp. 721-725.)—Since the value of quicklime depends on the quantity of CaO present, a method for determining this is proposed, which is based on the fact that the alkaline earths form definite compounds with sucrose. With CaO at least three are known, containing respectively one, two, and three molecules of CaO to one of sucrose, the two first being formed by dissolving CaO in the cold in a sucrose solution, while the third is precipitated on warming the solution.

The preliminary experiments cited confirm Lamy's* observation that the solubility of CaO in sucrose solution varies inversely as the temperature, and prove that not less than 150 c.c. of a 10 per cent. sucrose solution are required to dissolve the CaO in one gramme of quicklime. Other ordinary constituents of quicklime, such as iron, alumina, magnesia, silica, and calcium carbonate are either quite insoluble in the sucrose solution, or soluble only to an inappreciable extent. One gramme of magnesia, after being shaken with 150 c.c. of sucrose solution, showed only 0.001 gramme of MgO in the clear filtrate.

The following is the method adopted: One gramme of the finely-powdered substance is shaken for twenty minutes with 150 c.c. of 10 per cent. sucrose solution, the liquid filtered, and the clear solution titrated with standard HCl, half an hour being sufficient for the whole operation. Test samples of quicklime, containing very little CaCO₃, were analysed by this method, and the results compared with those obtained in the usual way by precipitation as calcium oxalate.

Sample.	Quicklime taken. Grammes.	Volume of 10 per cent. sucrose solution taken.	Per cent. CaO by titration method.	Per cent. CaO by gravimetric method.	Variation of volumetric method.
1.	1.020	150	92.12	93.00	—0.88
2.	1.090	150	91.90	92.28	—0.38
3.	1.006	150	92.15	93.10	—0.95
4.	1.108	150	95.01	95.90	—0.89
5.	1.023	150	87.30	87.70	—0.40
6.	1.232	150	91.70	92.30	—0.60

The small quantity of CaCO₃ present would account for the discrepancy between the gravimetric and the sucrose methods.

C. A. M.

* *La sucrerie indigene et coloniale*, 11-19.

Method for obtaining Pure White Magnesium Pyrophosphate in the Citrate Method. Dr. Hugo Neubauer. (*Zeit. für angewand. Chem.*, 1894, Heft 22, pp. 678, 679.)

In the estimation of phosphoric acid by the citrate method, it is often almost impossible to obtain the precipitate perfectly white even after long-continued ignition. This may be remedied by the addition of ammonium sulphate (5 grammes) before precipitating with the magnesia mixture. When heated for five minutes over a strong flame, the precipitate becomes perfectly white. Any precipitate of calcium sulphate formed by adding the ammonium sulphate may be ignored, since CaSO_4 is readily soluble in ammonium citrate.

C. A. M.

A New Method of Estimating Milk-fat. Dr. P. Fernandez-Krug and Dr. W. Hampe. (*Zeit. für angewand. Chemie*, 1894, Heft 22, pp. 683-687.)—In this process a measured volume of the milk is mixed with a finely-divided mineral substance, and the water removed by adding another finely-powdered material which combines with it without affecting the milk-fat. The dried mass is shaken out with ether, and the dissolved fat estimated by evaporation of a definite portion of the solution.

The following are the details of the actual estimation: About 7.5 grammes of washed and dried kaolin are introduced into a nickel basin holding about 200 c.c., and made into a creamy paste with 5 c.c. of the milk. This is then well mixed with about 5 grammes of finely-powdered anhydrous sodium sulphate, which combines with the water in the milk. A fine state of division is necessary to prevent a crystallization of the sulphate with the water, which would cause a loss of milk-fat. The dried mass is placed in a flask holding about 100 c.c. and closed with a well-fitting stopper, and 25 c.c. of ether added. After shaking for five minutes and cooling to the temperature of the air, 5 c.c. of the clear liquid are removed by means of a pipette, the point of which is covered with cotton-wool to prevent particles entering, and the dissolved fat estimated by evaporation in a weighed flask. From this the percentage on the whole can be calculated. The results obtained by this method agree well with those obtained by the older method, in which sand is mixed with the milk and the fat subsequently extracted with ether. Results in grammes per litre:

	Full Milk.	Full Milk.	Watered Milk.	Skim Milk.	Skim Milk.
Old Method ...	34.6	36.0	21.2	2.7	3.2
New Method ...	34.6	36.0	21.2	2.9	3.3

When analysing butter-milk or sour milk it is necessary to have them as homogeneous fluids. To effect this, a litre of the butter-milk or sour milk is warmed to about 45°, shaken for five minutes with 10 grammes of NaHCO_3 , and allowed to cool to the temperature of the room. The authors promise their results as soon as they have proved that, besides fat, no other constituents of the neutralized milk dissolve in ether.

The contribution concludes with a description of an apparatus by means of which,

from the weight of the fat, its percentage in the milk can be immediately read off. It is on the principle of the steel-yard with a running weight, the mass of which is calculated for the specific gravity of the milk and other conditions. C. A. M.

On the Estimation of Nitrogen in Nitro-cellulose. Duschon Stanojewitsch. (*Zeit. für angewand. Chemie*, 1894, Heft 22, pp. 676-678.)—Lunge's method of estimation by measurement of the gas liberated by the action of H_2SO_4 and mercury on the substance is open to the objection that the action of the air in the flask on the nitric oxide causes an error. To avoid this, the author has devised an apparatus which combines the methods of Schloosing and Lunge.

The weighed substance is introduced, together with 50 or 60 grammes of mercury, into the decomposition flask, which is connected with a modified nitrometer filled with a 25 per cent. solution of soda. After the air in the flask has been displaced by a current of CO_2 , 10 to 15 c.c. of H_2SO_4 are introduced, and the flask shaken until all the gas is liberated. To completely drive it over into the nitrometer, the flask is warmed while a current of CO_2 is passed in. For measurement it is advisable to introduce the gas into a measuring cylinder filled with water, which can be done by means of a delivery tube with a stop-cock at the top of the nitrometer. The apparatus is especially suitable for the estimation of nitro-glycerine in dynamite. Minute pieces of the sample are digested in the flask with H_2SO_4 and mercury. The decomposition is rapid, and the presence of carbonates does not influence the result. In the case of gelatinized nitro-cellulose it is necessary to warm the mixture over a water-bath, but the mercury and nitro-cellulose should not be warmed before the introduction of the H_2SO_4 , since the reaction is then so violent that an explosion may occur. C. A. M.

A New Method of Estimating Glucose by Alkaline Copper Solution. MM. Allein and Gaud. (*Jour. Pharm. et Chim.*, 1894, xxx., pp. 305-307.)—To avoid errors introduced by the base keeping the copper in solution, the authors recommend the substitution of ammonia for the fixed alkalies. To cause ammonia to act on glucose it must be kept in a sealed tube with it at 100° for thirty or forty hours, and even then only a small quantity of the glucose is destroyed, 5 to 6 per cent. of formic acid being produced; whereas glucose, treated in a similar way with the fixed alkalies, yields up to 60 per cent. of lactic acid.

The solution is prepared by dissolving 8.7916 grammes of electrolytic copper in 98 grammes of H_2SO_4 , diluting with an equal quantity of water and making up to a litre with concentrated ammonia solution. 10 c.c. of this is equivalent to 0.05 gramme of glucose. Being free from organic impurities, the solution keeps well.

In the estimation, 10 c.c. of the copper solution are measured into a flask and 10 c.c. of ammonia solution added. The flask is furnished with a cork with three holes, so that the extremity of the burette containing the sugar solution can be inserted and a current of hydrogen introduced. It is placed on a water-bath maintained at a temperature of from 90° to 95° while the hydrogen is passed in. When the temperature

of the interior of the flask is at 80° , the sugar solution is introduced drop by drop from the burette until the copper solution is decolorized. Cuprous oxide is formed, but is dissolved to a colourless liquid at the moment of formation by the ammonia, so that it is easy to judge when the blue colour is about to disappear. Excellent results are said to be obtained by the process, the only disadvantage of which is the necessity of manipulating at from 80° to 85° in order not to drive off the ammonia.

C. A. M.

On the Estimation of Boron. Henri Moissan. (*Bull. Soc. Chim. de Paris*, 1894, 18-19, pp. 955-958).—The process is a modification of Gooch's method, by which the estimation is made more rapid and loss by volatilization avoided.

The weighed boron compound, which must be in the form of boric acid or a borate, is placed in a distilling tube, the neck of which is closed by a stoppered funnel ground into it. One c.c. of HNO_3 is introduced, and the contents of the flask distilled to dryness by means of a water-bath. The distillate, condensed by the usual spiral worm, falls into a flask containing an excess of ammonia and connected with bulb tubes also containing ammonia. The residue in the flask is distilled with successive portions (10 c.c.) of pure methyl alcohol, being taken to dryness each time, until the alcohol distils over free from boric acid, as shown by the flame test. The ammoniacal distillate is emptied into a platinum basin, in which is a known quantity of calcium hydrate, previously weighed in the anhydrous state. On evaporation over a water-bath at about 70° , borate of calcium is left, and the increase of weight gives the quantity of boric anhydride from which the boron in the sample can be calculated. The calcium oxide is prepared by calcining pure calcium nitrate. A large excess is necessary: for 0.5 grammes boric acid, about 8 or 10 grammes.

The method gave the following results when used to estimate boric anhydride prepared from a pure boric acid:

I.	II.	III.	IV.	
99.73	99.67	99.62	99.6	per cent.

The author has used the process for the estimation of boron in the phosphate and sulphate of boron and in metallic borates and borides.

C. A. M.

Toxicological detection and estimation of Arsenic. Ernest Barillot. (*Bull. de la Soc. Chim. de Paris*, 1894, Tomes xi. and xii., pp. 958, 959).—Marsh's process is modified so that the arsenic deposited in the tube may be weighed. The generating flask is connected with a tube of green glass heated in three sections by burners. The substance to be analysed, after the animal matter has been destroyed by one of the ordinary processes, is treated with fuming nitric acid and potassium nitrate, the nitrous acids being afterwards removed by heating with excess of sulphuric acid. The solution is concentrated down to about 200 c.c., and after being proved to be free from nitric and nitrous acids, is introduced gradually into the generating flask, where the gas is liberated in the usual manner. The sections in which the rings of arsenic are deposited are detached and weighed, washed with fuming HNO_3 , dried and again weighed, the difference giving the amount of arsenic.

C. A. M.

Separation of Copper from Cadmium, Zinc and Nickel. P. N. Bikowa. (*Chem. Zeit.*, 1894, xviii., 1789).—The author utilizes the reaction given by a soluble iodide and phenylhydrazine with a copper salt (see preceding abstract) to separate copper from cadmium, zinc and nickel. The solution containing the metals is acidulated with sulphuric acid, treated with phenylhydrazine (the hydrochloride may be used, as the presence of a chloride is of no significance in this use of the reaction) and precipitated with potassium iodide, added drop by drop, to avoid an excess, in which the precipitate is somewhat soluble. Provided enough phenylhydrazine be present, a small excess of potassium iodide is not detrimental. The copper is completely separated, and the remaining metals are retained in solution. The remaining stages of the determination may be performed by the ordinary methods. Salts of mercury and iron must be absent, as they yield precipitates with phenylhydrazine.

B. B.

The Purification of Saltpetre from Perchlorate. V. Panaotovič. (*Chem. Zeit.*, 1894, xviii., 1567).—The author confirms the statements of previous observers that commercial saltpetre is usually contaminated with perchlorate (*vide* THE ANALYST, xix. 22). Out of 180 casks from a well-known Hamburg firm, 122 contained more than 0.25 per cent. of perchlorate, and only 7 were perfectly free therefrom. Although saltpetre containing perchlorate must have been used for years in powder manufacture, yet it does not necessarily follow that the presence of this impurity is a matter of indifference. Explosions that have received no satisfactory explanation may have been due to it, the more so as pure potassium perchlorate, when substituted for potassium chlorate in the manufacture of signal-lights, has proved unstable, spontaneous ignition taking place after four months' storage. These facts are sufficient to render it desirable to remove perchlorate from saltpetre intended for the manufacture of explosives. An attempt to effect this by fusing the salt until the perchlorate had been decomposed proved a failure, as much nitrite was formed, together with some free alkali, the removal of which products was difficult and costly. The purification was eventually achieved by dissolving the saltpetre in hot filtered spring-water (free from chlorine) to a strength of 48° B, adding a little size to favour the separation of insoluble impurities, decanting and allowing the solution to cool to 25° C, stirring to promote the separation of small crystals. The mother liquor (28° B) was poured off, and the crystals washed five or six times with pure water. The mother liquor was concentrated, and the separated crystals re-treated. From the final mother liquor crystals of potassium perchlorate were isolated and identified.

B. B.

Preservation of Starch Solution. H. Král. (*Pharm. C. H.*, 1894, xv., 606; through *Chem. Zeit.*)—The author recommends that the starch solution used in carrying out Hübl's iodine absorption process be prevented from decomposing by the addition of chloroform. A small quantity of chloroform is added, and the mixture well shaken. The product can be kept for months without suffering change.

B. B.

Detection of Nitrites in presence of Iron Salts. H. Král. (*Pharm. C. H.*, 1894, xv., 619.)—The detection of nitrites by means of an iodide and starch solution—*e.g.*, in drinking-waters—fails in the presence of ferric salts, as the latter also liberate iodine. The acid necessarily added is a source of error, even when acetic acid is used, as iodine is slowly liberated in the absence of nitrites. These drawbacks can be avoided by the use of oxalic acid, in the presence of which ferric salts are stated to liberate no iodine. B. B.

REVIEW.

THE SALE OF FOOD AND DRUGS ACTS. BELL and SCRIVENER. Second edition. (Shaw and Sons, Fetter Lane, E.C. Price 4s. 6d.)

The appearance of a second edition of this book affords additional evidence of the increased interest taken in the working of the Food Acts. The chemical reviewer would be guilty of a presumptuous act were he to venture upon a criticism of the purely legal portions of a book written by two learned barristers, but as far as we can see, the authors have recorded, in a very systematic manner, the most important of the numerous decisions given in courts of law touching the Sale of Food and Drugs Acts. Perhaps we might have been spared a good many of the decisions of the lower courts, which, as every analyst knows, are puzzling and contradictory.

But the authors have attempted to do more than write a legal guide, for, apparently without any chemical qualification, they have ventured to enter into the discussion of matters purely chemical, with results such as might have been anticipated. In commenting upon Section 6, truly described as "a most important section, and the entire working of the Sale of Food and Drugs Act very nearly depends upon it," the authors discuss the composition of the more important articles of food. We are sorry to find that the remarks under this head are neither characterized by the absence of bias nor by the presence of modern knowledge. They often appear to be a faint echo of the information contained in a well-known little handbook on "The Chemistry of Foods" published eleven years ago. Thus, the remarks as to the variability of the composition of butter-fat are obviously not based upon analyses of commercial butters made from the mixed milk of a number of cows. In connection with the adulteration of butter, "the more common offence" is, nowadays, not the sale of a factitious article, but rather the sale of mixtures of butter and margarine. The remark that "it is a common practice to mix a certain proportion of starch with yeast as a preservative" appears uncalled for, unless the book were written for defence advocates only. As to milk, the book takes us back to those bygone times in which the notorious lecture was delivered at the Health Exhibition, the Manchester milk case occurred, and the old Wanklyn process was in use, with limits fixed at 9 per cent. and 2.5 per cent. respectively.

The chemical part of the little work affords another proof of the good sense contained in the old adage, where the cobbler is advised to strictly confine his attention to his last. O. H.

THE ANALYST.

FEBRUARY, 1895.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE Annual General Meeting of the Society was held at Burlington House on Wednesday, January 16. In the unavoidable absence of the President, Mr. Otto Hehner occupied the chair.

The minutes of the previous meeting were read and confirmed.

Mr. Herbert Stephen Shorthouse, 47, Pershore Road, Birmingham, was elected a member of the Society.

The accounts for the year, which had been audited by Mr. Kitto and Dr. Teed, were presented by the Treasurer, Mr. E. W. Voelcker. These showed that the Society was in a more satisfactory financial condition than in any previous year.

Referring to that portion of the accounts connected with THE ANALYST, the Sub-Editor, Dr. W. J. Sykes, said they had again had a successful year. Since the journal had been in the Society's hands, the receipts in connection with it had risen steadily and continuously, whilst the working expenses had diminished.

On the proposition of Dr. Stevenson, the report and accounts as presented were unanimously adopted.

On the motion of the chairman, a vote of thanks was passed to the President and Council of the Chemical Society for having allowed the Society the use of their rooms during the past year.

The valedictory address of the retiring President, Sir Charles A. Cameron, M.D. (whose duties as Medical Officer of Health detained him in Dublin), was then read by Dr. Bernard Dyer, Hon. Secretary.

RETIRING ADDRESS OF THE PRESIDENT, SIR CHARLES A. CAMERON, M.D.

GENTLEMEN :—Those who contend that the offices of Public Analyst and Medical Officer of Health should not be combined in the same person, may fairly claim to have, in my absence from the annual meeting of the Public Analysts' Society, a strong proof of the soundness of their views. In my capacity as President of the Society of Public Analysts, it is clearly my bounden duty to be present at their annual gathering. On the other hand, as the Medical Officer of Health for Dublin, it is imperative that I should be at my post whenever an epidemic is present. Unfortunately this is the case just now, for small-pox, absent for fourteen years from Dublin, is now spreading rapidly throughout the city. Whilst no member of our Society will suffer in health by the absence of the President from the annual meeting, the presence of the Dublin

health officer at his post at this juncture might possibly be the means of preventing some people from contracting one of the most loathsome of diseases. May I venture, therefore, to hope that my absence from your annual meeting will not be regarded as a proof of want of interest on my part in the affairs of our Society, but that it is due to unavoidable circumstances? I feel indeed most grateful to you for having allowed me to occupy for the period of two years the distinguished position of your President. Your kindness in this respect I regard, not merely as a compliment to myself, but also as one to the section of our Society who reside in Ireland. Realizing how little I have done to merit being placed in this position, I feel all the more sensible of the kindness shown to me by one and all of our members.

I feel under a great obligation to the ex-President, who has so kindly acted as my substitute during the last two years—I refer to my friend, Mr. Otto Hehner. I may without flattery say of him that the substitute was much superior to the person whose place he occupied in his knowledge of the affairs and objects of our Society.

I am rejoiced to think that I transfer the Presidency of our organization to a distinguished chemist of whom we all feel proud. He enters upon his duties at a time when our Society has reached the highest degree of prosperity to which we have yet attained. We are more numerous than we have ever before been. Our members are to be found throughout all the English-speaking countries. Our work compares favourably with that of a similar kind performed in any other country. The interest felt in our meetings during the year seems to have been fully maintained, and the papers read at them, together with the discussions which followed, have not fallen below the standard quality.

THE ANALYST, under its new management, exhibits a decided improvement, which is all the more remarkable, seeing that the cost of producing it has been decreased.

Our members have no doubt been much interested in the proceedings of the Committee of the House of Commons, who were appointed last session to inquire into the workings of the Sale of Food and Drugs Act. Up to the present the evidence submitted to the Committee has not on the whole been in favour of enacting a more stringent measure to abate adulteration. The Public Analysts have, however, yet to be heard, and we must hope their evidence will convince the Committee that to relax the provisions of the present rather weak Acts would not be to the advantage of the public at large, though it might benefit a few individuals. I trust that the outcome of the Committee's deliberations will be the adoption of a Bill substantially the same as that drawn up recently by our Society. I think that every effort should be made to induce the Committee to recommend the fixing of standards for all articles that admit of it. In prosecutions for excess of water in butter, the defendant's counsel or solicitor constantly raises the point that there is no legal standard in the case, and that consequently the Public Analyst has no right to fix one. The immense experience which the Public Analysts have in reference to the composition of food, their general scientific knowledge, and, I may add, their common-sense, are sufficient to enable them to determine what ought to be the maximal and minimal proportion of any of the variable constituents of food. We are not indebted to the dairy farmers for our knowledge of the minimal amount of non-fatty solids in milk. We cannot rely upon

them to tell us what is the maximal quantity of water which might fairly and properly be permitted in butter. For my part, I shall continue to hold that no butter, fresh or salted, need contain more than 16 per cent. of water. If through carelessness or want of skill larger quantities should be found, then it is the maker, and not the consumer, who should suffer. The following statistics in reference to the amount of water in Irish butter may prove interesting:

In my laboratory in 1893-94, 1,434 specimens of butter were examined, of which 503, or 35.1 per cent., contained more than 16 per cent. of water. In 1893, 549 specimens were examined. They came from the four Provinces, but those from Munster contained the largest proportion of water, as shown by the following table:

	Munster.	Leinster.	Ulster.	Connaught.
Number of samples	357	67	79	46
Percentage containing less than 16 per cent. of water	50.10	85.07	86.07	76.08
Percentage containing more than 16 per cent. of water	49.90	14.93	13.93	23.92

In 1894 the figures stood as follows:

	Munster.	Leinster.	Ulster.	Connaught.
Number of samples	604	179	50	52
Percentage containing less than 16 per cent. of water	57.61	86.60	92	82.70
Percentage containing more than 16 per cent. of water	42.39	13.40	8	17.30

Out of 961 specimens from Munster, 22 contained less than 9 per cent. of water; whilst in the 473 specimens from the rest of Ireland there were 43 containing less than 9 per cent. of water. No fewer than 87 of the 961 Munster specimens contained more than 20 per cent. of water, whilst in the 473 samples from the rest of Ireland, only 5 included more than 20 per cent. of water.

The largest percentage of water, namely, 34, was found in a Munster specimen.

In 1894 the percentage of water in the Irish butter that came into my laboratory exhibited a great improvement as regards excess of water as compared with 1893. This improvement may be due to the comparative coolness of 1893 as compared with the previous year. To some extent it perhaps may be accounted for by the great number of prosecutions for excess of water instituted in Ireland, chiefly in Munster. In the county Clare, for which I am Public Analyst, there were 70 successful prosecutions for excess of water in the half-year ended December 31, 1893. Within the last two years nearly 200 convictions have taken place on my certificate, nearly all in Munster. The magistrates who tried the cases were resident or stipendiary magistrates and justices of the peace. Many of the latter being country gentlemen, and farming more or less, were butter-makers themselves, yet with hardly an exception they adopted the statement in my certificate that 16 per cent. of water was the maximal amount that should be tolerated in butter.

A short time ago the decision of a Limerick Petty Sessions Court inflicting a fine for excess of water in butter was reversed by the County Court judge (Mr. Adams). This judge, who has a great reputation for wit, said that chemists were about the least reliable of witnesses!

Let us now return to the affairs of our Society.

Death was not very busy in our ranks. In 1894 our death-rate was only 0·8 per 100 of our members, or less than half the rate for the whole population. We have lost only two members, Dr. C. R. Alder Wright, F.R.S., and Dr. Emmerson. Dr. Wright, although not a Public Analyst, was a chemist and analyst of high reputation, and he took a great interest in the affairs of our Society. His loss to science will long be felt. Dr. Emmerson was one of our oldest members, and rendered good services in the districts (Leicestershire, Rutland, Northampton, Peterborough, etc.) for which he was Public Analyst.

In addition to our losses by death, we have had a few secessions and removals of members; but, on the other hand, our gains have exceeded our losses. In 1894 one honorary and ten ordinary members were elected, together with four associates. At present our Society is constituted as follows:

Honorary members	10
Members	210
Associates	31
Total	251

No fewer than twenty-four papers were read at the monthly meetings, and whilst all were more or less interesting, several of them were very valuable contributions to analytical chemistry. The following is a list of the papers read in 1894:

"Milk and Milk Products." By H. Droop Richmond.

"Note on the Detection of Cotton-seed Oil in Lard." By E. J. Bevan.

"The Loss of Total Solids in Milk on Keeping." By E. J. Bevan.

Notes from the Laboratory of the Royal Agricultural Society of England: (i.) "The Occurrence of a Poisonous Leguminous Seed in Indian Peas. (ii.) "Analyses of Waters from Wells in close proximity to Churchyards." By J. Augustus Voelcker, Ph.D., B.Sc.

"Some Frequently Neglected Errors of Analysis." By H. Droop Richmond.

"On Beef Stearin in Lard." By Otto Hehner.

"Note on Lemon and Orange Peel." By E. G. Clayton.

"Note on the Analysis of Phosphor Tin." By Frank L. Teed, D.Sc.

"A Simple Apparatus for the Extraction for Analysis of Gases dissolved in Water." By Sidney Harvey.

"On the Recognition of Exhausted Ginger." By A. H. Allen and C. G. Moor.

"On the Change in the Composition of Butter by Long Keeping." By A. H. Allen and C. G. Moor.

"The Examination of Urine for Sugar." By Alfred H. Allen.

"The Composition of Cheese." By W. Chattaway, T. H. Pearmain and C. G. Moor, B.A.

- "Note on the Valenta Acetic Acid Test." By W. Chattaway, T. H. Pearmain, and C. G. Moor, B.A.
- "The Detection of Methylated Spirit in Tinctures, Spirits, and other Compounds." By A. Ashby, M.B., F.R.C.S.
- "Roasted Chicory." By E. G. Clayton.
- "The Need for Fuller Statistics of Adulteration." By C. H. Cribb, B.Sc.
- "Note on Woody Fibre Determination." By A. P. Aitken, D.Sc.
- "Note on the Analysis of Sodium Peroxide." By L. Archbutt.
- "Note on Gladding's Process for the Determination of Resin in Soap." By L. Archbutt.
- "Note on a Sample of Red Water." By W. F. Lowe.
- "On Rechnagel's Phenomenon (the Rise in the S. G. of New Milk)." By H. Droop Richmond.
- "The Composition of Milk, and the Conditions Affecting it, as shown by Dr. Bell's Analyses." By J. F. Liverseege.

The preparation of a suggested Bill for the amendment of the existing Acts in relation to adulteration occupied no inconsiderable proportion of the time of our members who undertook the task. It is to be hoped that the Bill, or something closely resembling it, may be adopted by the Committee of the House of Commons, to whom it will be submitted. I would venture to suggest one amendment to the existing Acts, namely, that the Public Analyst should not be required to receive the whole of a sample of which the vendor has refused a part. When the inspector purchases or procures a sample, he ought to divide it at once into two parts; if the vendor refuses to accept a portion, one part should be sent to the analyst, and the other, sealed up at once, should be retained by the inspector for production in court. There appears to be no object gained by the analyst, and not the inspector, dividing the sample, except that unnecessary trouble is imposed upon the analyst. Of course this observation does not apply to the cases referred to in clause 15 of the suggested Bill. With respect to the transmission of articles to the analyst, the Irish Court of Queen's Bench have decided that they may be sent by railway. Bulky boxes of samples of beer, milk, etc., cannot well be sent by post.

It would be desirable to provide that when a mixture of an article with one much cheaper is sold, some indication as to the relative proportion of each constituent of the mixture should be given to the purchaser. I once certified that a mixture of 95 parts of chicory and 5 of coffee was not of the nature, substance, and quality of the article demanded. The magistrate who tried the case held that so long as there was any coffee in the mixture, it could not be regarded as an adulterated or debased article.

It is satisfactory to find that the principal recommendations of our Society for the amendment of the anti-adulteration Acts were approved of by the Congress held last autumn in London under the auspices of the British Institute of Public Health.

In bringing this short address to a conclusion, I must thank most heartily my friend Dr. Dyer, one of our energetic and zealous secretaries, for having during the last two years kept me posted up on all the affairs of our Society. I earnestly hope

that he and all the members of our Society may enjoy full measures of happiness and prosperity in 1895, and in many a succeeding year.

Mr. Hehner proposed that a very hearty vote of thanks be accorded to the retiring President for his interesting and valuable address, and for his services during his Presidency.

Dr. Stevenson, in seconding Mr. Hehner's proposition, which was carried with acclamation, said that the absence of Sir Charles, although much to be regretted, was easily excused under the circumstances.

Dr. Rideal and Mr. Cribb having examined the ballot-papers, the Chairman announced that the following gentlemen had been elected officers and Council for the ensuing year :

President—Thomas Stevenson, M.D., F.R.C.P.

Vice-Presidents (who have filled the office of President)—M. A. Adams, F.R.C.S. ; A. H. Allen ; Sir Chas. Cameron, M.D., F.R.C.S. ; A. Dupré, Ph.D., F.R.S. ; Otto Hehner ; Alfred Hill, M.D. ; J. Muter, Ph.D., M.A., F.R.S.E. (Who have not filled the office of President)—A. Wynter Blyth, M.R.C.S. ; Walter J. Sykes, M.D. ; John A. Voelcker, M.A., B.Sc., Ph.D.

Treasurer—E. W. Voelcker.

Hon. Secretaries—Edward J. Bevan, Bernard Dyer, D.Sc.

Other Members of Council—Charles E. Cassal, W. Chattaway, C. H. Cribb, B.Sc., W. W. Fisher, M.A., B. Kitto, H. Droop Richmond, F. W. Stoddart.

The names of those Members of Council whose term of office has not yet expired, and who consequently do not retire this year, are James Baynes, Sidney Harvey, W. Morgan, Ph.D., W. Cobden Samuel, and W. F. Keating Stock.

Dr. Thomas Stevenson, the newly-elected President, then took the chair amidst loud applause, and said that he desired to return his hearty thanks for the honour done him. He felt the position a somewhat difficult one, since he could not pretend to the qualifications which the late President possessed. Mr. Hehner had referred to the foundation of the Society in terms which brought many old friends back to his mind. It might not be known to all that he (Dr. Stevenson) was the first Treasurer of the Society. His mind went back twenty years, for he found that the first general meeting took place on February 5, 1875. He should endeavour always to be present at the meetings, though he could not promise never to fail, and to contribute as much as possible to the interests of the Society.

In the absence of the author, Dr. Dyer read the following paper :

NOTE ON THE USE OF MAIZE AS AN ADULTERANT OF OATMEAL.

By JOHN WHITE, F.I.C.

In the course of my work under the Sale of Food and Drugs Act, I have recently had submitted to me a large number of samples of fine oatmeal. Upon making a microscopical examination of these in the ordinary way, I noticed in several the presence of some granules larger than those of the oat, which, upon further investi-

gation, proved to be maize. This is to me an entirely novel adulterant of oatmeal, and I cannot discover any definite record of its previous use. The fact, moreover, that it does not appear to have been certified as an adulteration by any Analyst—at any rate recently—is my reason for bringing it before the Society.

There is of course no new chemical process to be described in this connection, but as the detection and estimation of a facettad starch such as maize, in the presence of oatmeal, is much more difficult than the recognition of a non-facettad starch like barley, a short description of the method I adopted may be useful to others.

The whole of the sample should be thoroughly mixed in a large mortar. Then take a small quantity, from 0.25 to 0.5 of a gramme; rub down in a small ground-glass mortar with from 5 to 10 c.c. distilled water, and while the particles are in suspension, take out a drop on the blade of a penknife and place on the slide. The estimation may now be made by comparing with standard mixtures put up in a similar manner. The micrometer may, of course, be used with advantage; but the most valuable information as to the amount of maize present is obtained by examining under polarized light, the black cross being shown perhaps better by this starch than by any other. I have found that an occasional oat granule will polarize, but the cross is naturally very much smaller than the one shown by maize and not so well defined, and very little practice is necessary to enable one to distinguish between the two with certainty. I obtained this result with oats ground by myself, and which had been supplied to me by the grower.

The amount of ash does not seem to give any clue to the presence of maize. In some of the adulterated samples it was rather higher than the average, but not abnormally so.

In considering the likelihood of maize becoming a regular adulterant of oatmeal, it is necessary to compare their respective market values. At the present time the two cereals are about equal in price in the raw state, and the vendor of one of these samples raised this point in his defence at the hearing. It must, however, be borne in mind that the proportion of meal to husk in maize is very much higher than in the oat, and consequently, equal weights of raw material being taken, much more maize-meal than oatmeal will be produced.

In neither of the samples was the amount of maize present large, the maximum I have yet found being 3 per cent. In some of the samples there were traces of barley starch in addition.

DISCUSSION.

Dr. Dyer said it was singular that the proportion of maize should be so small as 3 per cent. Of course, in a sample of home-grown oats the presence of maize could scarcely be accidental, but with foreign oats it was by no means remarkable. A great deal of grain of all kinds, including oats, was imported from maize-growing countries, and very often the quantity of total admixture or foreign matter was very considerable. Oats, for instance, were deemed commercially pure if the total amount of admixture was within 3 per cent. The foreign matter, which often amounted to very much more, consisted not merely of earth and chaff, but of other grains, including barley,

wheat, rye, etc., and occasionally a few grains of maize, so that small quantities of foreign starches might very well be found in the meal. He did not know if oats of this class were used for grinding; they were no doubt chiefly used for horses; but the facts he pointed to should lead analysts to be cautious.

Mr. Cassal said he had recently examined some official samples of oatmeal adulterated with barley. He had certified two samples to contain 10 and 15 per cent. of barley respectively. The adulteration of oatmeal with barley-meal was no doubt remunerative, but 3 per cent. was a curiously low percentage in a matter of this particular kind.

Dr. Dyer remarked that barley was a very cheap grain indeed. Foreign barley was somewhat dearer now, but during the last year could have been bought at 11s. a quarter. He, too, had examined samples of oatmeal adulterated with barley, in which the percentage of barley was considerably in excess of that reported by Mr. Cassal.

Mr. Cassal said that Mr. Hehner, who examined some of his samples, arrived at practically the same conclusion as himself. His authority did not prosecute in these cases.

Dr. Dyer said that the prices of grain fluctuated a great deal, and were different for home produce and for foreign imported grain. The price of malting-barley was high, but grinding-barley was cheap—only about one-half or one-third of the price of malting-barley, and much cheaper than good Scotch oats.

The two following papers were read by their respective authors :

NOTE ON A SAMPLE OF SUPPOSED LARD.

By BENEDICT KITTO, F.I.C.

LARD, being a substance which easily lends itself to various forms of sophistication, has from time to time given public analysts a considerable amount of work; and it is no doubt advisable that any peculiarity observed in samples should be brought to the notice of the members of our society. It was suggested to me by our former president (Mr. Hehner) that a recent experience of mine should be so notified.

A sample of "lard" was submitted to me for analysis, under the Sale of Food and Drugs Act, which, when subjected to the iodine absorption test, was found to take up 14.96 per cent. only of iodine. Having thus found that it was not pure lard, Reichert's test was applied, and the $2\frac{1}{2}$ grammes operated upon required 4.4 c.c. of decinormal alkali. These results at once pointed to cocoa-nut oil. The sp. gr. at 100° C. was .868, and the melting-point 27.5°. The potassic hydrate neutralized was 22.95 per cent., showing a saponification equivalent of 244.

Having no cocoa-nut oil by me for comparative tests, I went round to Mr. Hehner's laboratory, and he very kindly supplied me with a deodorized sample, and, I may add, ever willing to place his great experience at the service of his brother analysts, offered to go through the iodine absorption and Reichert's tests on a

portion of the sample, at the same time pointing out to me the harshness observable upon pressing the knife against the cocoa-nut oil, as if it had a crystalline structure. This feeling was also very marked when I tried my own sample. Mr. Hohner's tests confirmed my own. There was a taste noticeable in the sample which I could not recognise, but no distinct smell.

Although most of the tests corresponded fairly well with the results obtained from the cocoa-nut oil, the iodine absorption was much higher, owing probably to the admixture of a small proportion of cotton-seed oil—Becchi's silver nitrate test having pointed, though not conclusively, in that direction—or possibly to a little lard oil.

I reported the sample to be almost entirely composed of prepared cocoa-nut oil, and therefore not lard at all; and I believe this was not disputed in court. A conviction of the seller followed.

So far as I know, this is the first case for some years where cocoa-nut oil has been detected doing service in this direction. About six years ago, Mr. A. H. Allen communicated to this society a case in which he found 33 per cent. of cocoa-nut oil as an adulteration in lard (ANALYST, xij., 189); but I have not heard of its having been noticed since that time until this sample was analysed.

NOTE ON A SAMPLE OF LARDERINE.

By R. BODMER.

IN November last I received from one of my inspectors a sample labelled "Lardarine." The article had been exposed for sale as lard, but before completion of the purchase by the inspector the vendor declared it to be "lardarine"—a word coined in imitation of margarine.

The sample contained a trifling proportion of water. On analysis the following figures were obtained:

Melting-point	38° C.
Sp. gr. at 100° F. (water at 100° F. = 1)	0.9105.
Reichert (2.5 grammes)	1.0 c.c. $\frac{N}{16}$ Baryta.
Temperature reaction (Maumené)	43° C.
Iodine absorption	65.6 per cent.
Saponification 1 gr. required	201.8 mg. KHO.
Saponification equiv.	277.7
Nitrate of silver	copious reduction.
Crystallization from ether	beef crystals.
Ether deposit (Stock's process)	37 mg.
Oleo-refractometer	+6°*

The sample was evidently a mixture of beef stearin, cocoa-nut fat, cotton-seed oil, and *probably* some lard.

Taking the Reichert figure as indicating about 25 per cent. cocoa-nut fat, the presence of which is also confirmed by the saponification figure, and the E. W. D.

* Kindly taken for me by Mr. Bevan.

showing about 10 per cent. of beef stearin, and working out the iodine absorption, the following approximate proportions are obtained :

Beef stearin	10 per cent.	} Giving a calculated iodine absorption of 65 per cent.
Cocoa-nut fat	25 "	
Cotton-seed oil	45 "	
Lard	20 "	

Thus :

10 per cent. beef-stearin	=	2.5	iodine.
25 " cocoa-nut fat	=	2.25	"
45 " cotton-seed oil	=	48.15	"
20 " lard	=	12.00	"

64.90

I understand that lardine is now a recognised article of commerce, and the question naturally suggests itself whether the Margarine Act should not be extended so as to make it a penal offence to expose for sale a spurious lard without the same being labelled "Lardine," or some such term.

DISCUSSION.

Mr. Richmond asked Mr. Bodmer to say what strength of acid was used in his Maumené test, and how it was done, so that they might compare it in their own minds with their own ways of doing it. He would also like to have some figures obtained by the same strength of acid for one or two well-known oils, such as olive oil and cotton-seed oil.

Mr. Cassal wished to know what Mr. Bodmer meant by "beef crystals." There might be different opinions as to the characters of such crystals, and some views as to those characters which had been prematurely accepted were erroneous. It was useless, for instance, to examine the crystals obtained by a third crystallization.

Mr. Hohner said that lard substitutes had been sold for many years without any secrecy, to which, if they were sold under their proper titles, there could not be any reasonable objection.

This sample was a peculiar one ; its iodine absorption was lower than is usual with lard substitutes, and it had apparently been made up with the object of giving a normal iodine absorption. He doubted whether Mr. Bodmer's sample really contained lard, because it seemed somewhat unreasonable to put much lard into a mixture sold as a lard substitute.

The other sample, which he had had the privilege of seeing, was very much like pure cocoa-nut oil with a very small admixture of other substances. Purified cocoa-nut oil was well known ; but he had never before heard of its being used or sold almost pure for lard. It seemed, however, to be a promising lard substitute, and, personally, he should prefer a pure vegetable substance to a mixture of doubtful composition.

Mr. Bodmer said he performed the Maumené test with half the quantities recommended by Mr. Allen, because the samples he received were usually small. As a rule the results were comparable with those obtained by Mr. Allen. The rise in temperature was 40° C. for olive oil, and about 75° C. for cotton-seed oil.

His statement as to beef stearin must be taken with a certain amount of faith. The crystals he obtained were distinctly plumose, like those obtained from actual beef stearin. The amount of ether-washed deposit corresponded, according to Stock's table, with about 10 per cent. of beef stearin. From the melting-point and the iodine number it was pretty certain that beef stearin was present.

Mr. Hehner remarked that beef stearin would undoubtedly be present; in fact, it was a necessary constituent of such mixtures.

Mr. Cassal said he was not expressing doubt as to the presence of beef stearin in the sample. He wanted to know upon what evidence the statement was founded. Plumosity alone was not sufficient evidence to prove the crystals to be beef crystals.

After the meeting the annual dinner took place at the Criterion, Piccadilly.

THE DETERMINATION OF WOODY FIBRE IN FODDERS.

By A. P. AITKEN, D.Sc.

(Read at the Meeting, Dec. 5, 1894.)

THE name woody fibre is a conventional one, including under it a considerable number of substances, which agree in this—that they are composed of carbon, hydrogen and oxygen, and are insoluble in dilute solutions of acid and alkali when boiled in these for a certain time.

The generally recognised method of estimating woody fibre is to boil the finely-ground substance in a solution of $1\frac{1}{2}$ per cent. sulphuric acid for half an hour, maintaining the solution at the same strength all the time. The substance is then washed till quite free from acid, and thereafter boiled for half an hour in a $1\frac{1}{2}$ per cent. solution of caustic soda maintained at a constant strength, and after thorough washing the residue is dried at about 110° C. and weighed. This substance contains a certain amount of ash, and usually a small amount of albuminoid matter, both of which are determined and their weight deducted. The residue is regarded as woody fibre. This is, of course, a purely conventional method, laying no claim to scientific accuracy, nevertheless yielding information which is of some value in determining the purity or quality of a fodder, and is of some use as a rough guide in determining its feeding value.

My object in the present note is not to discuss the value of the method itself, or the information which may be obtained by means of it, but simply to direct attention to a modification in the practical carrying out of the process that will be found to make it simpler and less irksome, and to yield results that are more uniform than those usually attained. There is no item in the analysis of a fodder wherein analysts differ so widely as in the woody fibre. It is no uncommon thing to find in two analyses of the same fodder by different analysts that the one has found double the amount of woody fibre that the other has when the total amount is comparatively small.

This may arise from their using different strengths of acid and alkali, or from their continuing the boiling for a longer or shorter time. Since the quantity of woody

fibre found varies according as the solutions are stronger or the times of boiling are longer, this is one of those matters upon which analysts should come to an agreement and adopt a prescribed method of working, so that the results obtained may at least be comparable, and the simpler the method prescribed, the more likely is it to be adopted.

Some analysts use 5 per cent. solution of acid and alkali, and some prefer $2\frac{1}{2}$ per cent. solutions; but $1\frac{1}{4}$ per cent. solutions are more convenient, as they are less liable to frothing and more easily washed out of the residue. The results obtained by using stronger solutions are in most cases less than those obtained by using the weaker ones, but the weakness of the solutions used can be compensated by increasing the duration of boiling. The maintenance of the strength of the solutions during boiling is achieved either by adding water from time to time as they evaporate, or by attaching the flask in which the process is carried out to a return-flow condenser. When a large number of estimations are going on at once, neither of these methods is convenient, and when flasks are used in which to conduct the operation, the frothing of the contents causes much trouble. This is got over, or at least lessened, by introducing a blast of air into the flasks, but that also is attended with inconvenience. I get over the difficulty entirely by abandoning the use of flasks altogether, and by abandoning the boiling also. I now use beakers, which I immerse in a steam-bath, and which I cover with watch-glasses. The result is that the solutions are maintained at the temperature of boiling water and no evaporation takes place, so that the strength of the solutions does not alter, and the subsequent washing of the residues and their final removal from the beaker to a filter or Gooch crucible is easy. The extraction of the soluble matters is not as rapid when the solutions are simply subjected to the temperature of the steam arising from boiling water, but the difference is not very great—three-quarters of an hour in the steam-bath is equivalent to half an hour's boiling. During the operation the solutions require no attention, there is no frothing, no evaporation, no breaking of flasks, as is not unfrequent with the boiling process. The bath I use is simply a water-bath of elongated shape, at the bottom of which is a gutter kept automatically full of water, and only about an inch deep. The water is kept constantly boiling by means of little jets of gas issuing from a number of small holes bored in the top of a horizontal piece of brass tubing extending the whole length of the gutter, and fixed below it at a distance of about an inch and a half. The gas-jets are about two inches apart, and less than an inch high. The lid of the bath is removable, and is perforated with holes, into which the beakers are let down and suspended by their rims, and the bottoms of the beakers do not quite reach the level of the water-gutter. The whole body of the beaker is thus immersed in steam at about 100° C., and the contents of the flasks are subjected to perfectly uniform conditions, so that successive determinations of woody fibre so conducted give results of great uniformity, and are attended with less trouble than the boiling methods commonly practised.

In the case of oil-cakes, it is advisable to extract the oil with ether before setting about the determination of the woody fibre. A sufficiently complete removal of the oil can be effected in a very short time, as the small residue will be saponified during the alkali treatment.

Unless the bulk of the oil is removed before treatment, the fibre left after treatment with acid and alkali will contain a considerable residuum of oil; if, therefore, the oil contained in the sample operated upon has not been extracted before the fibre determination, the fibre obtained must have the oil extracted from it before weighing.

DISCUSSION.

Mr. Allen remarked upon the laxity which existed in speaking of the strengths of acids and alkalies. Percentages by volume and percentages by weight were often confounded, which led, of course, to grave errors and misunderstandings. Thus, if sulphuric acid of $2\frac{1}{2}$ per cent. by weight were prescribed, and the operator used water containing $2\frac{1}{2}$ per cent. by measure, he would be employing an agent containing about $4\frac{1}{2}$ per cent. by weight of the acid, and hence of nearly twice the prescribed strength.

Mr. Sidney Harvey agreed with Mr. Allen as to the importance of correctly and clearly defining the strength of solutions. He had for many years adopted the plan suggested by Dr. Aitken for heating, and his results were generally very fairly comparable.

Dr. Dyer said that in his own laboratory the custom was to boil up the material in a beaker over a low flame, from time to time adding a little water from a wash-bottle when the liquid boiled down, a mark on the side of the beaker serving as a guide. Although a crude way of work, it answered very well if it was watched, but attention was necessary to prevent any adherence of fibre to the sides of the beaker, which was apt to cause something like caramelization, leading, if unnoticed, to discrepant results. He thought, however, that errors were more usually caused by incomplete extraction of the oil from the fibre, either before or after treatment with acid and alkali. Mere alkaline treatment failed to saponify more than a fraction—sometimes only a moderate fraction—of the oil.

The Preparation of Litmus Solution. W. Schafer. (*Apoth. Zeit.*, 1894, ix., p. 839; through *Chem. Zeit.*)—Litmus solution of good quality can be prepared as follows: The dye stuff in cubes, as commonly sold, is placed in a percolator and extracted with distilled water. The extract is evaporated until it equals the weight of the original litmus, then treated with three times its weight of 90 per cent. alcohol, made strongly acid with hydrochloric acid and allowed to stand for two days. Azolitmin is precipitated in brown clots, while the accompanying dirty-violet colouring matter remains dissolved in the alcohol. The precipitate is collected on a filter and washed two or three times with acidulated hot water, until the filtrate, which is faintly red in colour, becomes on treatment with ammonia pure blue, without any violet tinge. The purified azolitmin thus obtained is brought into solution in water made faintly alkaline with ammonia, and the solution is diluted with distilled water until it is equal to three and a half times the weight of the original litmus. It is then accurately neutralized and preserved by the addition

of 10 per cent. of alcohol. The litmus solution thus prepared leaves nothing to be desired, its change of colour, when used as an indicator, being perfectly sharp.

B. B.

The Concentration and Distillation of Hydrogen Peroxide. R. Wolffenstein. (*Ber.*, 1894, xxvii., 3307; through *Chem. Zeit.*)—According to the experiments of the author, hydrogen peroxide is much more stable at moderately high temperatures than is generally supposed. The instability of this substance, which is often observed, must be referred to the presence of certain definite impurities. In order to secure its stability, hydrogen peroxide must be free from any substance capable of giving an alkaline reaction, from all traces of compounds of the heavy metals, and from solid substances of all kinds, even those usually accounted chemically indifferent. Distillation experiments conducted with hydrogen peroxide, complying with these requirements, show that the vapour is stable at moderate temperatures. Moreover, a 3 per cent. solution of hydrogen peroxide may be concentrated to about 45 per cent. without any considerable quantity volatilizing. When, however, this concentration is reached, the rate of volatilization rapidly increases, until at a pressure of 68 mm. and a temperature of 85° C., a product which is perfectly pure and almost anhydrous passes over. Hydrogen peroxide thus obtained is a clear, syrupy liquid, evaporating when exposed to air, and giving rise to a stinging sensation and the formation of white spots when brought in contact with the skin. It is *acid* to litmus, although dilute solutions are neutral to that indicator. The simplest method of preparing pure hydrogen peroxide from the 3 per cent. solution commonly sold consists simply in distilling the entire quantity. When larger quantities of more concentrated solution are required, the commercial peroxide (3 per cent. strength) is concentrated on the water bath to about 20 per cent.—care being taken that the preparation is free from the impurities mentioned above—and continuing the concentration *in vacuo* up to about 50 per cent., extracting with ether, and finally distilling *in vacuo*. By fractional distillation, hydrogen peroxide of any desired concentration may be thus obtained.

B. B.

Fallacies of Post-Mortem Tests for Morphine. Daniel L. Davoll, jun. (*Jour. Amer. Chem. Soc.*, 1894, xvi., No. 12, pp. 799-808.)—The experiments here detailed were made to determine to what extent cadaveric alkaloids might respond to the usual tests for morphine. For this purpose the internal organs of a dog, which had been buried in a tight pine-box for forty-five days, were extracted by a modification of the older method, and the extract tested with the ordinary morphine reagents.

Bromine water, Wagner's solution, Mayer's reagent, and sodium phosphomolybdate all gave strong precipitates, and a cloudiness was obtained on the cautious addition of ammonium hydrate. The most satisfactory tests were Fröhde's reagent, Le Fort's test, and ferric chloride, all of which gave a negative result with the pure extract, while they would detect as little as 0.00005 gramme of added morphine. Of the three, Fröhde's reagent was the most sensitive. Iodic acid gave

indications of a reducing agent in the extracted matter, and HNO_3 and ferric chloride with potassium ferricyanide were quite useless.

During the process of extraction of the different organs a decided odour, resembling that of tri-methylamine, was given off.

C. A. M.

On the Gunning Method for Total Nitrogen in Fertilizers. W. E. Gargigues. (*Jour. Amer. Chem. Soc.*, 1894, xvi., No. 12, pp. 795-799.)—In the estimation of fertilizers containing nitrates the Gunning method, as ordinarily applied, gives too low results. This is clearly shown in the following determinations, for which a mixture of pure sodium nitrate and tankage was prepared. The latter was valued by the ordinary Kjeldahl and Gunning methods and by soda-lime combustion with the following results. Percentage of nitrogen :

Kjeldahl.	Gunning.	Soda-lime.
7.05	7.05	7.08

The nitrogen in the mixture was then determined, and gave :

Theory.	Kjeldahl.	Gunning.
8.97	8.90	7.17
		7.31
		7.46

The author's explanation of the lower results obtained by the Gunning method is that the potassium sulphate, being added at too early a stage, protects the sample from the attack of the acid.

To avoid this, the following method of proceeding is recommended : The salicylic acid mixture is first added, and when the sample has been thoroughly wetted, the flask is allowed to stand for fifteen minutes, after which the thio-sulphate, is added in four or five portions with constant shaking. Each portion should be completely dissolved before the next is added. The potassium sulphate is then introduced, and the whole digested as usual.

When treated in this way the above-mentioned mixture gave :

8.85 8.87 8.90 8.92 per cent. of N.

Other mixtures gave equally satisfactory results, but in the case of sodium nitrate alone, neither by the Gunning nor the Kjeldahl method could a sufficiently accurate determination be obtained. A carefully prepared sample of the salt containing by theory 16.49 per cent. of nitrogen yielded :

Kjeldahl.	Gunning.
16.05	15.80
16.35	16.00
	16.15
	16.22
	16.38

No means of avoiding this loss could be found, and it would thus appear justifiable to take the highest figure in closely agreeing duplicates, instead of the usual average.

To avoid loss by frothing in the distillation, it is advisable to use a 32-ounce wash-bottle, instead of the 500 or 600 c.c. flask. C. A. M.

[EDITORIAL NOTE.—The Kjeldahl-Jodlbauer-Gunning method, skilfully worked, gives without difficulty *the whole* of the nitrogen in nitrite of potassium or sodium, or even nitrate of ammonium, whether alone or in admixture with other forms of nitrogen found in fertilizers. The sulphonated salicylic acid (or phenol) mixture must be poured *rapidly* on to the substance, after which reduction is performed with zinc dust gradually in the cold first, and then with but very gentle heat, till all zinc is dissolved. A drop of mercury is desirable, but not absolutely necessary. The whole is boiled for a time, after which, *and not till then*, the sulphate of potassium is added.—B. D.]

The Determination of Melting-Points and the Composition of some Candle Material. W. E. Garrigues. (*Jour. Amer. Chem. Soc.*, 1894, xvi., 12, pp. 825-832.) —For the purpose of obtaining concordant results by different observers, a modification of Dalican's method of taking the solidification point is recommended. 30 to 50 c.c. of the dried sample are placed in a small beaker, the thermometer bulb inserted in the melted mass, and the glass and its contents turned in one direction until the mercury stops receding or ascending. With stearic acid there is a rise of about 0.3°, and the temperature remains constant for about a minute. With paraffin wax there is no rise, but the mercury remains constant about half a minute, and then recedes at the rate of about 0.1° to 0.2° per minute. With mixtures of the two there is great diversity, ranging from no rise at all to one of nearly 4°. The latter represents a mixture of $\frac{2}{3}$ stearic acid to $\frac{1}{3}$ wax. The following table gives the melting-point and behaviour of the mercury in the case of mixtures in different proportions:

Stearic Acid.	Paraffin.	Melting-Point. Degrees.	Mercury. Degrees.
100	0	54.6	0.1 rise
100	10	53.4	No rise
100	20	52.4	"
100	30	51.6	"
100	40	50.8	0.05 rise
100	50	50.1	0.2
100	60	49.2	0.6
100	70	48.2	0.8
100	80	47.4	2.8
100	90	45.6	0.6
100	100	45.2	0.8
90	100	45.8	No rise
80	100	46.3	
70	100	46.9	
60	100	47.3	
50	100	47.9	
40	100	48.6	
30	100	48.9	
20	100	49.6	
10	100	50.4	
0	100	50.8	

In cases where no rise of the mercury takes place, the twirling should be continued until a homogeneous semi-solid consistency is attained. If still no rise occurs, the temperature at which there was a pause is noted.

The latter part of the paper is chiefly concerned with the changes which take place in the distillation of the fatty acids of the fats used in candle-making. The conclusion arrived at is that the stearic and palmitic acids distil without change, while the oleic acid partly splits up into liquid hydrocarbons, tar, and a saturated or non-iodine consuming fatty acid.

C. A. M.

On the Technical Analysis of Asphaltum. Laura A. Linton. (*Jour. Amer. Chem. Soc.*, 1894, xvi., 12, pp. 809-822.)—It is usual to designate the constituents of bitumens as "Petrolene" and "Asphaltene" according to their behaviour with different solvents. The determination of these is purely empirical, but for want of a more scientific method is employed in this paper. In general terms, *asphaltene* is that portion of different forms of bitumen which is soluble in CS_2 , chloroform, benzene, and a few other less well-known liquids, and is not soluble in the menstrua that dissolve petrolene. *Petrolene* includes a large number of hydrocarbons, and is much less definite in character than asphaltene. Ethyl ether and the so-called petroleum naphtha are the solvents generally used for it. Of twenty-three asphalts examined in their relation to the solvents, turpentine, CS_2 and chloroform, all yielded some asphaltene to chloroform after previous treatment with hot turpentine; and in several CS_2 following turpentine dissolved a considerable quantity. It was found that Californian naphtha and ethyl ether could not be used as solvents for petrolene, since they also dissolved some of the asphaltene, and petroleum ether was therefore used.

The following method of analysis is recommended: Two portions of the asphalt are weighed in Erlenmeyer flasks; 50 c.c. of petroleum ether are added, and next morning decanted on a counterpoised filter placed in a three-inch funnel with a stopcock in its neck. The residue in the flask is digested with successive portions of petroleum ether until the liquid decanted through the filter ceases to be coloured. The whole of the bitumen is then transferred to the filter, washed well with petroleum ether and dried, together with the counterpoised filter, in a steam bath. The loss in weight of the bitumen represents the petrolene. The flask should also be dried and weighed, and any increase in weight deducted from the amount determined as petrolene. To determine the asphaltene, the substance on the filter is digested firstly with successive portions of hot turpentine, and secondly with chloroform, until the filtrates are colourless. During the digestion, the stopcock in the neck of the funnel is kept closed. The flask should also be rinsed out with the same solvents, and the washings poured over the filter. The loss in weight after drying the latter represents the asphaltene.

The filter is then burned in a platinum crucible, and the loss in weight represents the organic matter not bitumen.

The method is capable of yielding closely concordant duplicate results at each step. A typical analysis gives :

Water.	Petrolene.	Asphaltene.	Other Organic Matter.	Mineral Matter.	Total.
2.029	32.4455	22.1115	8.1215	35.2865	99.994.

C. A. M.

On the Chemical Composition of Cola-nut. C. Uffelmann and A. Bömer. (*Zeit. für angewand. Chemie*, 1894, xxiii., pp. 710-712.)—The mean results of the analysis of ten samples of Cola-nut, varying in price from 4s. to 20s. per pound, were :

Water.	Total Nitrogen.	Protein.	Caffeine (including Theobromine).	Ethereal Extract.
13.35	1.53	5.91	2.08	1.35
Starch.	Tannin.	Cellulose.	Other Extracted Matter (non-nitrogenous).	Mineral Matter.
45.44	3.79	7.01	18.21	2.9

In spite of the great difference in price, the different specimens showed a remarkable similarity in composition.

Of the inferior plants used to adulterate Cola-nut, only two, according to the analysis of Heckel and Schlagdenhauffen, contain caffeine, viz., *Cola Ballay* nut, 1.05 per cent., and the nut of *Cola gabonensis*, 0.263 per cent. The other usual adulterants are quite free from the alkaloid.

C. A. M.

Contribution to Milk Analysis. Ernst Beckmann. (*Report of Thirteenth Assembly of the Bavarian Association of Chemists*, 1894, pp. 16-18.)

(a) *Estimation of Fat.*

The disadvantages of the usual methods of extraction may be avoided by the following process, the object of which is to remove only such a quantity of the water that the ether can readily dissolve all the fat, but not so much that an emulsion is formed. This is most readily effected by precipitation of the casein and fat by means of lead acetate, and separation of the greater part of the serum by filtration.

Twenty-five c.c. (or, in very accurate determinations, a weighed quantity) of the milk are diluted with an equal volume of water in a stoppered glass cylinder. To the mixture 2.5 c.c., or, in the case of sour milk, up to 5 c.c., of official lead acetate solution* are added, the cylinder well shaken, and, finally, sufficient NaHCO_3 solution introduced to cause the precipitate to coagulate. After filtration, the residue on the paper is placed in a glass cylinder, together with small glass balls of about 3 mm. diameter, and shaken out with ether. The object of the glass balls is to prevent an emulsion, and to facilitate the solution of the fat in the ether. The filter-paper is also shaken out with ether in a second cylinder, and the solution added to the other one. The dissolved fat is then estimated either by distilling off the ether or by separating it by dilution. The process is rapid, and is equally reliable with every description of milk.

* One part lead acetate, three parts litharge, and ten parts water.

The following table gives the percentage of fat found in different specimens of milk, with control results by the Soxhlet method :

		New Method.	Soxhlet.
(1)	Fresh milk	4.065	4.07
(2)	Fresh skim milk	0.91	0.85
(3)	Sour milk	2.93	2.99
(4)	" "	2.92	2.88 and 3.02
(5)	" "	3.65	3.65
(6)	Sour skim milk	1.07	0.970

(b) *Freezing-Point.*

Since the freezing-point of a fluid depends on the nature and quantity of the substances dissolved in it, and not on those of the bodies in suspension, the determination of the freezing-point of milk will furnish a check result influenced by the amount of water present, but not by that of the fat. The normal freezing-point of milk is very constant, ranging from -0.54° to -0.58° , the mean being -0.554° . The lowering of the freezing-point below that of water is in proportion to the concentration of the milk.

The determination is made by placing the milk in a suitable vessel, surrounded by a mixture of ice and salt, inserting a thermometer, and constantly stirring until the solidification point is noted. At first the temperature falls from one to two degrees below the freezing-point, but when the separation of ice commences, rises again, and remains constant for some time. This is taken as the point of solidification. By the use of a thermometer graduated in one-hundredths of a degree, the reading is simple, but since an addition of 10 per cent. of water to a milk only alters the freezing-point by five and a half hundredths of a degree, it is to be feared that such a difference is too small to draw a conclusion from when the results are not concordant. In the case of sour milks and milks to which salts have been added, the depression of the freezing-point is increased, but in such cases the simultaneously increased electrical conductivity will correct the conclusion.

The author hopes to produce a freezing apparatus in a handy form, to serve as a check in milk analysis.

C. A. M.

On a Method of Estimating Sulphocyanic Acid. M. T. Goudoin. (*Journ. de Pharm. et Chimie*, 1894, ii., pp. 481-484.)—The acid is estimated as cuprous sulphocyanide, care being taken that no free HNO_3 is present. The liquid, if alkaline, is made acid with SO_2 solution (density, 1005), and a 10 per cent. solution of pure CuSO_4 added so long as a precipitate forms. It is then heated at 80° until the blue tint appears, when the precipitate is filtered on a tared filter, washed till no longer acid, dried at 100° , and weighed.

The author's experiments prove that the cuprous sulphocyanide is stable in the presence of the two reagents, CuSO_4 and SO_2 ; and that the method is applicable in the presence of all salts usually met with in industrial waste waters. In a test mixture containing CaCl_2 , NaCl , Na_2CO_3 , MgCl_2 and 0.10 per cent. of potassium sulphocyanide, a precipitate of cuprous sulphocyanide was obtained corresponding to (1) 0.1004 per cent. and (2) 0.0994 per cent. of the potassium salt.

Cyanides do not interfere with the result.

C. A. M.

Estimation of Gelatin and Albumin in Peptone. Ernst Beckmann. (*Report of Thirteenth Assembly of Bavarian Chemists*, 1894, pp. 18-20.)—A method for the estimation of gelatin in meat extracts is based upon the fact that formaldehyde combines with it to form a non-fusible and insoluble compound—formalin-gelatin. In order to render insoluble one gramme of gelatin dissolved in water, about two drops of the 40 per cent. solution in water of formaldehyde (formalin) are added. The quantity required is so trifling that its weight may be neglected.

The presence of much free acid hinders the reaction, which, however, takes place perfectly well in a slightly alkaline solution :

- (1) 0.235 gramme gelatin, dissolved in a little water, furnished 0.209 gramme formalin-gelatin.
- (2) 0.274 gramme gelatin, dissolved in a little water, furnished 0.231 gramme formalin-gelatin.

If formic or acetic acid be present, either too little precipitate is obtained, or none at all.

- (1) 0.1840 gramme gelatin in 10 c.c. H_2O , treated with ten drops of formic acid, and left in the water bath ten minutes, yielded 0.0866 gramme.
- (2) 0.1256 gramme gelatin, on adding ten drops acetic acid with the formalin, yielded 0.0496 gramme insoluble residue.
- (3) 0.1420 gramme with 1 c.c. acetic acid gave no insoluble residue with formalin.

Soluble egg-albumin and serum albumin left residues on mixing with formalin.

- (1) 0.3680 gramme soluble egg-albumin gave 0.3408 gramme residue.
- (2) 0.3040 gramme soluble serum albumin gave 0.2810 gramme residue.
- (3) A mixture of 0.308 gramme gelatin and 0.2524 gramme egg-albumin gave 0.5402 gramme insoluble residue.

Merck's dry peptone was found to be completely soluble in the presence of formalin, and by this means gelatin and albumin could be easily separated from peptone.

- (1) A solution containing 0.294 gramme gelatin and 0.49 gramme peptone gave 0.256 gramme residue.
- (2) A solution containing 0.272 gramme serum albumin and 0.557 gramme peptone gave 0.265 gramme residue.

In determining whether a meat-extract contains gelatin, the albumin is estimated in an aliquot part of a watery solution by means of acid. Another portion is treated with formalin, steamed on the water bath, and, after boiling for a short time with water, the residue is collected in a Gooch's crucible, dried at $100^{\circ} C.$, and weighed. After subtracting the amount of albumin previously found, this gives the gelatin. The peptone, etc., in the filtrate can be precipitated in the usual way. The method will be of use in milk analysis for detecting adulteration with an emulsion of gelatin and fat.

C. A. M.

Oils. G. de Negri and G. Fabris. (*Zeit. für Analyt. Chemie*, 1894, Heft 5, pp. 547-574. *From the Italian.*)—The contribution contains the quantitative figures and details of the colour reactions of olive-oil and forty different kinds of seed-oils.

Of the 213 samples of olive-oil examined, the authors obtained 160 from the fruits, the remainder being procured from reliable manufacturers. In pressed-out oils the melting-point of the fatty acids varied from 24° to 27° C. In extracted oils, which also contained more stearin, it lay between 25° and 29° C. The iodine number was between 79.5 and 88 per cent., and the saponification equivalent between 185 and 196. In no case did Becchi's test give any colour with pure olive-oil, which had been repeatedly filtered. The general conclusion arrived at is that no single test is sufficient to decide whether all olive-oil is pure. In certain specimens of olive-oil traces of arachidic acid are said to have been found.

In the case of earth-nut oil no colour reaction could be obtained by Becchi's, Milliau's, Baudouin's, or Schneider's tests, but the rapid solidification of the soaps in taking the saponification equivalent was not observed in any other oil.

In the application of Becchi's test for cotton-seed oil, the two solutions recommended by the Italian Commission were used :

(1) One gramme AgNO_3 in 200 c.c. of 98 per cent. ethyl alcohol, 40 c.c. sulphuric ether, and 0.1 gramme HNO_3 .

(2) Fifteen c.c. colza oil in 100 c.c. amyl alcohol.

Ten c.c. of the oil to be examined were mixed with (1) 1 c.c. AgNO_3 solution and (2) 10 c.c. of colza solution added. After being well stirred, the mixture was divided into two parts, one of which was heated for fifteen minutes in boiling water, the other being left cold.

With cotton-seed oleo-margarine no coloration was obtained by Becchi's or Milliau's reactions. The fatty acids melted at from 27° to 30° C.; the iodine number was 8.9 per cent.; and the saponification equivalent 257. The authors, therefore, conclude that its presence in butter and lard cannot always be detected. C. A. M.

On the Detection of Small Quantities of Metals of the Second Group in Precipitated Sulphur. B. Fresenius. (*Zeit. für Analyt. Chemie*, 1894, Heft 5, pp. 573-574.)—To the fluid containing the separated sulphur a layer of about 2 cm. of benzene or petroleum ether is added, the tube shaken well for a minute, and then allowed to stand for a minute. If the precipitate consists only of sulphur, the tube will contain two layers, the upper perfectly clear, and the lower very slightly turbid; but if traces of the metals of the second group are present, they will be observed as a thin coloured film at the junction of the two layers. C. A. M.

The Determination of Nitrogen in Nitrates, Nitro-compounds, and Nitroso-compounds. M. Krüger. (*Ber.* 1894, xxvii., 1633; through *Chem. Zeit.*)—0.2 to 0.3 gramme of the substance in which the nitrogen is to be determined is placed in a globular flask together with 20 c.c. of water (or an equal volume of alcohol if the

substance be soluble in that menstruum and difficultly soluble in water), and treated with 10 c. c. of an acid solution of stannous chloride and 1.5 gramme of spongy tin. The flask is then heated until the contents are colorless and the tin dissolved. (The alcohol, if present, is evaporated at this stage.) After cooling, 20 c. c. of strong sulphuric acid are added, and the mixture concentrated until fumes of sulphuric acid come off freely. When the liquid is again cool, potassium bichromate is added to oxidize amido-compounds, and complete resolution of the nitrogenous substances having thus been effected, the determination is finished in the usual manner. Substances which are volatile with steam even in acid solution and require the addition of alcohol, are thoroughly reduced by gentle warming on the water-bath until it is safe to evaporate off the alcohol in the manner mentioned above. A preferable plan, however, consists in carrying out the operation in a sealed tube immersed in a boiling water-bath.

B. B.

The Separation and Determination of Arsenic, Antimony and Tin. W. Hampe. (*Chem. Zeit.*, 1894, pp. 1900, 1901.)—The successful separation of these metals is always a matter of some difficulty, and depends in large measure on attention to details of manipulation rather than on the employment of any novel principle of separation.

Arsenic, antimony and tin are usually obtained in the course of an analysis as thio-salts, from which the sulphides can be recovered by precipitation with an acid. The precipitate of mixed sulphides, together with excess of sulphur, is dealt with in the following manner: The bulk of the precipitate is washed into a small beaker, and the residue on the filter is dissolved through by the least possible amount of the freshly-prepared ammonium sulphide into the same beaker. Whether the bulk of the precipitate dissolves or not is of no importance. The whole mass is treated with strong hydrochloric acid and oxidized with potassium chlorate; the chlorine is driven off, tartaric acid added, and the solution filtered. Excess of ammonia is added to the filtrate, which will remain perfectly clear, provided sufficient tartaric acid be present. More tartaric acid is needed when the mixture contains tin than when it contains only antimony. Arsenic is now precipitated by magnesia mixture from the solution kept at as low a bulk as possible, and the precipitated ammonio-magnesium arseniate is purified by solution in hydrochloric acid and precipitation with ammonia, the filtrate being added to the main filtrate, which is then acidulated with hydrochloric acid, and precipitated with sulphuretted hydrogen. From the mixed precipitate the sulphides of antimony and tin are separated in the following manner: The bulk of the precipitate is transferred to a beaker; the portion adhering to the filter-paper is dissolved through with freshly-prepared sodium sulphide used in as small quantity as possible, the washing being similarly restricted, so that the bulk of the solution in the beaker is but small. The total solution of sodium thio-salts is then treated with successive small quantities of sodium peroxide. It is to be noted that the sodium peroxide should be taken from the central parts of the tinplate canisters in which it is sold, as traces of tin from the metal of the canister might otherwise be introduced. As soon as the liquid has become colourless, and effervesces

on the addition of a further quantity of sodium peroxide, on account of the escape of oxygen, the oxidation of the thio-salts may be taken as complete, and the contents of the beaker then consists of caustic soda, sodium sulphate, sodium stannate and sodium antimonates. The solution is boiled, allowed to cool, and treated with one-third of its volume of alcohol of specific gravity 0.833 to precipitate the last traces of sodium antimonate. The beaker is roughly graduated as a guide to the addition of the proper quantity of alcohol. The solution is allowed to stand for 24 hours, and transferred to the filter with a mixture of one volume of alcohol and three of water, and washed according to Rose's prescription, namely, first with a mixture of one volume of water and one of alcohol of sp. gr. 0.833, and finally with one containing one volume of water and three of alcohol. All these washing liquids are made slightly alkaline by the addition of a drop of caustic soda. The sodium antimonate on the filter is dissolved in hydrochloric acid containing tartaric acid, the solution precipitated with sulphuretted hydrogen, and the antimony sulphide filtered and washed. As much as possible is detached from the filter and gently ignited in a stream of carbon dioxide and weighed as the anhydrous tri-sulphide; the residue adhering to the filter is dissolved through by means of ammonium sulphide, the solution evaporated to dryness in a weighed porcelain dish, and the residue oxidized with nitric acid ignited and weighed as Sb_2O_4 . The filtrate from the sodium antimonate is evaporated to drive off the alcohol, acidulated and precipitated with sulphuretted hydrogen. The tin sulphide is roasted to oxide and weighed. When a large quantity is present and the removal of alkali salts is consequently difficult, the precipitate is redissolved in ammonium sulphide and reprecipitated by sulphuric acid.

When a larger quantity of antimony than of arsenic and tin is present, as in hard lead, the antimony can be separated at the first stage of operations in the following way: The mixed sulphides are extracted with sodium sulphide and oxidized with sodium peroxide, the arsenic being converted into sodium arseniate and the sodium antimonate being precipitated. On endeavouring, however, to precipitate the arsenic in the filtrate from the antimony by means of magnesia mixture, it is found that the large quantity of sodium salts in solution prevents the complete precipitation of the arsenic. It is necessary therefore to evaporate the filtrate from the sodium antimonate to drive off the alcohol which it contains, then to precipitate the sulphides of tin and arsenic by acidulation with hydrochloric acid and by passing sulphuretted hydrogen, and finally to oxidize the sulphides with potassium chlorate and hydrochloric acid and separate them in the usual manner.

The method has been tried upon known mixtures and yields satisfactory results.

B. B.

Detection of Chlorine in presence of Iodine, and Quantitative Separation of these Halogens. P. N. Raikow. (*Chem. Zeit.*, 1894, xviii., 1661.)—The method is based on the formation of a precipitate when an aromatic hydrazine (*e.g.*, phenylhydrazine or tolylhydrazine) is mixed with copper sulphate, sulphuric acid, and a soluble iodide, and on the absence of such a precipitate when a chloride is substituted for the iodide. On treating an aqueous solution of the sulphate of phenyl-

hydrazine with one of copper sulphate, a bulky gray-brown precipitate is formed. Should either of the solutions contain free sulphuric acid, however, no precipitate is produced. When hydriodic acid or a soluble iodide is present, a voluminous precipitate, reddish-gray in colour, is thrown down. The test is carried out as follows: A freshly-prepared solution of the sulphate of phenylhydrazine is acidulated with dilute sulphuric acid, and treated with a solution of copper sulphate in excess. To the clear liquid the aqueous solution containing the chloride and iodide is added, and the mixture is shaken. A precipitate collects on the surface of the liquid, which appears clear and blue in colour. The precipitate is collected and washed with cold water. It contains the whole of the iodine. The chlorine may be recognised in the filtrate by means of silver nitrate after the addition of a further quantity of sulphuric acid, which is necessary to hinder the reducing action of the phenylhydrazine on the silver salt. The iodine is determined by boiling the precipitate with dilute caustic potash, filtering from copper oxide, acidulating with sulphuric acid, and proceeding in the usual manner. The bromine compound obtained by the use of phenylhydrazine is intermediate in solubility between the chloride and iodine compounds, and thus the presence of bromine interferes with the success of the separation.

B. B.

On the Detection of Geranium-oil in Rose-oil. Robert Jedermann. (*Zeit. f. Analy. Chem.*, 1895, p. 51.) As the result of an extended experience, the author has come to the conclusion that there is as yet no reliable test for the purity of rose-oil. Schiff's colour reaction and Hager's turbidity test are both unsatisfactory, and the freezing-point and specific gravity of the oil inconclusive. The specific gravity of rose-oil varies in different specimens according to the conditions of production and preparation. The pure stearoptene, which constitutes the scentless part of rose-oil, has a specific gravity of 0.813, while that of the oil free from stearoptene varies from 0.8730 to 0.8735. As the amount of this constituent in Balkan rose-oil varies from 7 to 30 per cent., and as the specific gravity of geranium-oil lies between 0.882 and 0.895, a rose-oil containing but little stearoptene, and consequently having a high specific gravity, is liable to be suspected of adulteration; on the other hand, the detection of very small quantities of geranium-oil is impossible.

The behaviour of the oil towards polarized light is also useless as a test, since rhodinol and geraniol are isomeric bodies, and behave similarly in this respect.

In consequence of the want of conclusive chemical and physical tests, the soap manufacturers and perfumers prefer to take the opinion of an expert who judges of the value of a sample by its odour.

C. A. M.

THE ANALYST.

MARCH, 1895.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday, February 6th, the President (Dr. Stevenson) occupying the chair.

The minutes of the previous meeting were read and confirmed.

Mr. D. J. O'Mahony, F.C.S., Public Analyst for the city of Cork and for the county of Cork, was proposed for election as a member of the Society.

Mr. Hehner then read the following paper :

ON THE DETERMINATION OF THE BROMINE ABSORPTION OF FATS GRAVIMETRICALLY.

By OTTO HEHNER.

It is not the writer's intention to add another to the already numerous modifications of processes intended for ascertaining the haloid absorption of unsaturated fatty compounds. The Hübl process, evolved from the bromine absorption process first described by Allen (*Pharmaceutical Journal*, September 25, 1880), and which is substantially the same as that used by Mills and Snodgrass (*Soc. Chem. Ind.*, 1884, No. 64), and Mills and Akitt (*Soc. Chem. Ind.*, 1884), is however, after all, merely a conventional one, and only gives concordant results when worked under exactly comparative conditions, from which, as laid down by Hübl, it is not desirable to depart. The process certainly does not measure the iodine absorption of any oil, but probably the iodochloro-absorption. From iodine solution, without the addition of mercuric chloride, as used by Gantter (*Zeitsch. f. Anal. Chem.*, 1893, p. 81), Dieterich, and others, oils take a far smaller percentage of iodine than corresponds to the Hübl number; and the proportion of mercuric chloride used has an immense influence upon the result. The present contribution is, therefore, merely intended as a study, and not as a proposed method, although I believe that in some cases it may be found both convenient, and that it may afford another factor in judging of the constitution of an oil.

The instability of the Hübl solution is a great drawback to its use. When many determinations are made this is not a matter of much moment, but when only occasional determinations are to be carried out the solution must always be freshly prepared and standardized; this gives an amount of trouble out of proportion to the result aimed at, and has the further disadvantage that freshly made solutions are apt to give capricious results.

In such cases a gravimetric method is always to be preferred to a volumetric one; the former has the additional advantage that the products of the reaction are obtained in such a form that their physical properties can be afterwards investigated, a matter, which, unfortunately, has been entirely neglected with the haloid addition products of oils, although promising valuable information.

If to an oil bromine is added, without the intervention of a solvent, combination takes place immediately; the reaction is, however, in many cases rather violent. By diluting with a suitable solvent, preferably chloroform or carbon tetrachloride, the action, though moderated, is equally complete.

A varying amount of hydrobromic acid is at the same time formed, and this is attributed by Mills to the presence of water in the materials; but is far more likely due to the formation of substitution compounds, besides the additive compounds. That this is so appears to me to be proved by some experiments which (following McIlhiney) I have made: Bromine added to chloroform containing some water produces no acid whatever, for when potassium iodide is added to such a mixture after a little time and in sufficient amount to convert the whole of the free bromine into a corresponding amount of iodine, and after the latter has been acted upon by an excess of hyposulphite solution, the resulting solution is perfectly neutral to phenolphthalein. This more or less roundabout method is necessary for testing, because iodine plus a hyposulphite yields tetrathionate and iodide without alteration of the reaction, while free bromine oxidizes the hyposulphite into sulphate, free sulphuric acid and free hydrobromic acid. The amount of free acid formed when bromine acts upon oil gives a measure for the amount of substitution. P. C. McIlhiney (*Jour. Am. Chem. Soc.*, 1894, p. 275; *ANALYST*, 1894, p. 141) has utilised in the manner previously employed and described by Allen (*Com. Org. Analysis*, ii., 383) the determination of the free acid formed as a measure of the substitution as distinguished from the addition. I have obtained the following figures: A sample of pure olive oil, treated with bromine, yielded 1.5 per cent. of free hydrobromic acid, another sample 2.7 per cent., castor oil 2.7 per cent., butter-fat 0.9 per cent., but a sample of boiled linseed oil 8.87 per cent. My figures, as far as they go, corroborate those found by McIlhiney, with the exception that he finds that no free acid is formed by the action of bromine upon boiled linseed oil. I know nothing about the origin of the sample tested by myself, and it is quite possible that it might contain either rosin or some other adulterant.

I operate as follows in order to obtain the bromo-compounds in a state fitted for weighing: A small, wide-mouthed fat flask is carefully weighed, and from 1 to 3 grammes of the fat to be tested introduced into it. These are dissolved in a few c.c. of chloroform, and then pure bromine is added, drop by drop, until the bromine is decidedly in excess. Both the chloroform and bromine must be previously tested in a blank experiment, to make sure that they contain no appreciable residue. The flask is then heated on the water-bath till most of the bromine is driven off; a little more chloroform is added, and the mixture again heated, the chloroform vapour helping to drive out the excess of bromine. The addition of chloroform may once more be repeated. The flask and its contents are then placed in an air-bath regulated for about 125° C., and kept there until their weight is constant; this takes

several hours. A little acrolein and hydrobromic acid escape during the drying, and the residue in some cases darkens slightly; in others a clear yellow heavy bromo-oil is obtained. Drying at 100° C. does not lead to satisfactory results.

The following are some of the results, as compared with the Hübl figures, upon the same samples. For comparison, by multiplying with 1.587, I calculate the gain in weight of the oil into iodine:

Substance.	Iodine by Hübl.	Iodine corresponding to Bromine gravimetrically.
Olive oil ...	80.3	81.5
" ...	80.2	79.9
" ...	80.6	80.7
Lard ...	65.7	64.4
" ...	"	64.6
" ...	63.2	64.1
" ...	60.1	61.4
Maize oil ...	122.0	123.2
Butter-fat ...	34.0	34.3
Mutton-fatty acids ...	48.1	47.8
Castor oil ...	83.0	69.5
Boiled linseed oil ...	132.5	159.5
Almond oil, fatty acids	—	102.3

In the case of the almond oil the Hübl figure was not determined, but calculating upon 95.5 per cent. of fatty acids in almond oil, the figure 102.3 for the fatty acids corresponds to 97.7, which is that given by the Hübl method for pure almond oil.

It will be seen that in most cases the iodine figure calculated from the gravimetric bromine absorption is in satisfactory approximation to the Hübl number, considering that that number is liable to variations of from 1 to 2 per cent., even in duplicate analyses (Amthor and Zink, *Zeitsch. Anal. Chem.*, 1892, p. 536). But in the case of castor oil the bromine process, as used by myself, gives a substantially lower result than the Hübl method; while in that of the sample of boiled linseed oil the reverse is the case. Whatever the explanation of this may be, and without desiring to generalize upon such scanty data, it is remarkable that in both those cases, where the oils contained more oxygen than ordinary oils do, the figures are substantially different. This difference may be worthy of further investigation. W. Fahrion (*Chem. Zeit.*, 1892, xvi., p. 1472) has already shown that the behaviour of castor oil towards Hübl solution is anomalous.

The numbers obtained by Mills, in concert with his two collaborators, in a few corresponding cases (*loc. cit.*), may be usefully cited here. I have calculated Mills's bromine figures into their iodine equivalents (80 Br : 127 I):

Substance.	Mills's Figure, as Iodine.	Average Hübl Number.
Almond oil ...	85.3	96—99
Maize oil ...	118.1	119
Olive oil ...	95.2	82—85
" ...	85.7	"
Castor oil ...	92.6	83—84
Butter-fat ...	44.3	26—38
Lard ...	59.2	50—67
Linseed oil ...	120.7	148—183
" (boiled)	162.6	—

Thus the Mills figures in almost every case agree very indifferently with the Hübl numbers. Curiously enough, Mills found a higher bromine number for castor oil than corresponds to the Hübl number; while for boiled linseed oil he finds actually a higher absorption than that of fresh linseed oil, when the contrary might be expected.

I would point out another curious fact, namely, that while unsaturated fats take up from an iodine solution, without the intervention of mercuric chloride, some iodine, showing that some iodo-addition compound is formed, it is not possible to obtain any gravimetric iodine addition number by acting upon such oils with iodine and drying the product. The iodine gradually volatilizes from the product, the weight of which decreases till it is not more, but sometimes even less, than that of the fat originally taken. The mass at first becomes green, then darkens much, almost chars at 125° C., and its melting-point rises considerably, solid fatty acids being formed. This is well known to be the case at very high temperatures, Wilde and Reichler (*Bull. Soc. Chem.*, 1889, No. 1, p. 295) having shown that oleic acid, heated with 1 per cent. of iodine to 270—280° C., yields about 70 per cent. of stearic acid. From my experiments it is obvious that this reaction takes place at much lower temperatures than these.

DISCUSSION.

Mr. Allen said he thoroughly agreed with Mr. Hehner that the reaction occurring in the Hübl method of determining iodine absorptions was far from being understood. He thought the investigation of the products formed in the process would afford new and important information. Thus if, after the titration was finished, chemists would take the trouble to evaporate the chloroform and weigh the residue, the weight of the residue would show whether the product was a chloro-iodo-derivative or an iodo-derivative pure and simple; though he gathered from Mr. Hehner's paper that iodo-compounds prepared in other ways were so unstable as to decompose with facility. The plan employed by McIlhiney of ascertaining the amount of hydrobromic acid formed, and hence deducing the extent to which substitution had occurred, was a method devised by him (Mr. Allen), and found very useful in examining the action of bromine on hydrocarbon oils. He congratulated Mr. Hehner in having hit on so short and easy a method of working, and should await with interest the further development of the process. Although he (Mr. Allen) was the originator of the idea of determining bromine absorptions, or, at least, the first to use and publish a workable process, the employment of the bromine in solution in a menstruum free from water was due to Mills and Snodgrass, whose method of working possessed distinct advantages. It was a remarkable fact that in certain cases the bromine absorption could not be calculated to the corresponding iodine absorption by the factor $\frac{127}{80}$. In 1886, the speaker pointed out the anomaly in the case of the results recorded for almond oil in 1886 (*Journ. Soc. Chem. Ind.*, vol. v., p. 282), and believed in this case some error must have existed in Mills' determination of the bromine absorbed.* But

* Mills and Snodgrass found the bromine-absorption of the fatty oil expressed from bitter almonds (from which the commercial oil of almonds is chiefly obtained) to be 26.3, while the oil from sweet almonds absorbed 53.7 per cent. of bromine.—A. H. A.

another curious anomaly exists in the case of linseed oil, which when raw was found by Mills and Snodgrass to take up 76.0 per cent. of bromine (=120.7 per cent. of iodine), whereas boiled linseed oil absorbed 102.4 per cent. of bromine (=162.6 iodine absorption). It was remarkable that a somewhat similar result should have been obtained by Mr. Hehner.

Mr. Richmond said he had had some experience with the Hübl process as applied to butter, and his figures were higher than the usual average. He frequently started the experiment at night, and made the titration the next morning, following in this Archbutt's recommendation. Somewhat higher results were obtained in this way than by allowing two hours for the action of the solution on the oil, the increase sometimes amounting to 4 or 5 per cent. in oils of high iodine absorption. It was very desirable that the process should be so arranged as to avoid such discrepancies if possible. If Mr. Hehner's process gave constant results—as it seemed to promise—it would be in many ways superior to the iodine absorption process.

The President said the paper was an extremely interesting one, especially in the close agreement of the figures, and the facility with which gravimetric measurements of the bromine compounds could be made. He hoped Mr. Hehner would be able to continue his investigations, and emphasized what Mr. Allen had said with regard to the desirability of examining the residues. He thought that the Hübl process might be satisfactorily worked by substituting a compound of iodine and chlorine for mercuric chloride.

Mr. Hehner, in reply, said it was regrettable that the haloid absorption processes had been worked volumetrically in every case; it seemed desirable to him that the products of the reaction should be studied as to their physical and other characters. The nature of the reaction involved in the Hübl process was far from clear; only very few, if any, figures obtained by the action of Hübl solution on pure unsaturated fatty acids had been published—indeed, it seemed exceedingly improbable that any pure unsaturated oleic acid had ever been prepared. When the formula of oleic acid was established nothing whatever was known of linoleic and linolenic acids, and the earlier investigators had, indeed, obtained their so-called oleic acid from linseed oil, which was now known to contain hardly any oleic acid at all. The explanation generally accepted as to the action of Hübl reagent was that the free iodine dislodged an equivalent amount of chlorine from the mercuric chloride, mercuric iodide and iodine chloride being formed; but if this were correct, there ought to be a separation of mercuric iodide, and this, as far as he knew, had never been observed. He thought the suggestion of the President as to dispensing with the use of mercury a valuable one, and he would try the use of a solution of iodine chloride instead of the Hübl solution. Mr. Richmond's remarks illustrated what he had said at the beginning of his paper, namely, that the tendency was for every chemist to make his own modification of the Hübl process, and as a consequence different workers often obtained widely divergent results, and this was hardly fair to the Hübl process itself.

Mr. Richmond then read the following three papers :

THE COMPOSITION AND ANALYSIS OF MILK AND MILK-PRODUCTS.

By H. DROOP RICHMOND.

THIS paper is a continuation of the annual reports of the work done in the laboratory of the Aylesbury Dairy Company. The results obtained in 1894 are given in the present communication. (For previous reports, see ANALYST, vii., 53; viii., 33; ix., 56; x., 67; xi., 66; xii., 39; xiii., 46; xiv., 69; xv., 44; xvi., 61; xvii., 62; xviii., 50; and xix., 73.)

The total number of samples analysed in 1894 was 32,295, consisting of

28,455 samples of milk.		2,269 samples of cream.	
706	„	separated and skimmed milk.	
174	„	butter.	18 „ buttermilk.
22	„	water.	38 „ sundries.
613	„	bacteriological examinations.	

Of the milk-samples, 12,633 were, in order to check the quality of the milk received, taken from the railway churns on their arrival at the company's chief depot. The bulk of the milk is distributed, with the least possible delay, to the customers, a certain portion being, however, utilized for the production of cream, etc. To control the men employed in delivering the milk, a further 13,361 samples were taken before, during, and after delivery, and analysed comparatively.

The fat has been estimated this year by the Leffmann-Beam method, and the total solids were calculated by the milk-scale.

AVERAGE COMPOSITION OF MILK DURING 1894.

Month.	On Arrival.				Before Delivery.	During Delivery.	After Delivery.
	Sp. Gr.	T. S.	Fat.	S.-n.-F.	T. S.	T. S.	T. S.
January ...	1·0322	12·84	3·98	8·86	12·74	12·80	12·75
February ...	1·0322	12·66	3·82	8·84	12·61	12·60	12·57
March ...	1·0322	12·57	3·74	8·83	12·55	12·50	12·53
April ...	1·0320	12·52	8·75	8·77	12·51	12·46	12·46
May ...	1·0323	12·47	3·66	8·81	12·44	12·41	12·37
June ...	1·0323	12·48	3·68	8·80	12·47	12·45	12·42
July ...	1·0319	12·44	3·74	8·70	12·43	12·38	12·36
August ...	1·0320	12·50	3·75	8·75	12·40	12·38	12·37
September ...	1·0322	12·62	3·81	8·81	12·57	12·51	12·52
October ...	1·0321	12·74	3·93	8·81	12·63	12·63	12·66
November ...	1·0322	13·14	4·24	8·90	12·95	12·97	13·04
December ...	1·0323	13·07	4·18	8·89	12·83	12·91	12·82
Average ...	1·0322	12·67	3·86	8·81	12·60	12·59	12·57

The figures differ but very slightly from those of last year. As is usual, the poorest milk is found in the summer, and the maximum is attained in November.

In comparing the different columns, it must be remembered that not only are

the samples taken at random, and are not directly comparable, but that, for the control of the business, more attention is naturally paid to milk the quality of which is known or suspected to be below the mean (*e.g.*, the morning's milk, which is always poorer than the evening's), than to that which is above the average. Allowing for this, the agreement of the averages is almost absolute.

Cream samples were taken before and during delivery. The average of the results is given in the following table :

AVERAGE AMOUNT OF FAT IN CREAM DURING 1894.

Month.	Before Delivery.			After Delivery.		
January	46·7	46·5
February	47·9	47·9
March	49·1	49·4
April	48·6	48·8
May	49·4	49·6
June	48·7	48·4
July	48·5	49·6
August	51·0	51·2
September	48·4	49·2
October	47·6	48·3
November	48·6	48·6
December	50·8	50·5
Average	48·9	49·1

Considering the difficulty of drawing average samples of cream of such richness the agreement between the two series is satisfactory.

The Leffmann-Beam method is now adopted also for cream analysis, with very satisfactory results.

The average composition of 44 samples of clotted cream is as follows :

AVERAGE COMPOSITION OF CLOTTED CREAM DURING 1894.

Water	31·59
Fat	60·25
Ash	·69
Solids-not-Fat	8·16

These figures are not greatly different from those found in former years.

The amount of fat in separated milk was in most cases less than 0·3 per cent., and on very rare occasions was it found to be above 0·4 per cent.

The composition of the butter analysed was as follows :

AVERAGE COMPOSITION OF BUTTER DURING 1894.

French Butter, fresh ; 41 samples.

Water	14·32 to 12·78	average	13·60
Fat	86·09 „ 84·19	„	85·05
Solids-not-Fat	2·47 „ ·78	„	1·35
Salt	·19 „ ·03	„	·09
Ratio of water to solids-not-fat, less salt	„	„	9·2

French Butter, salt; 31 samples.

Water	13.57 to 9.68	average	11.27
Fat	87.72 „ 82.98	„	85.28
Solids-not-Fat	4.63 „ 1.96	„	3.45
Salt	3.29 „ .57	„	1.97
Ratio of water to solids-not-fat, less salt	„	13.1

Brittany Butter; six samples.

Water	14.81 to 13.75	average	14.26
Fat	84.36 „ 83.18	„	83.84
Solids-not-Fat	2.01 „ 1.78	„	1.90
Salt25 „ .09	„	.16
Ratio of water to solids-not-fat, less salt	„	12.2

English Butter, fresh; 22 samples.

Water	15.23 to 11.90	average	13.49
Fat	87.62 „ 84.03	„	85.70
Solids-not-Fat	1.83 „ .48	„	.81
Salt	1.07 „ .01	„	.14
Ratio of water to solids-not-fat, less salt	„	5.0

English Butter, salt; 46 samples.

Water	16.39 to 10.19	average	13.11
Fat	87.65 „ 79.99	„	83.94
Solids-not-Fat	5.16 „ 1.90	„	2.95
Salt	4.30 „ .90	„	2.11
Ratio of water to solids-not-fat, less salt	„	6.4

Australian and New Zealand Butters, salt; 6 samples :

Water	12.53 to 8.24	average	10.72
Fat	89.82 „ 85.41	„	87.21
Solids-not-Fat	2.59 „ 1.44	„	2.07
Salt	1.55 „ .92	„	1.23
Ratio of water to solids-not-fat, less salt	„	7.9

An opportunity occurred for studying the change in composition in butter kept in casks. A salt butter was kept in a cask for one month, and was analysed directly after churning, and after one month :

	Water.	Fat.	S.-n.-F.	Salt.	Ratio of water to S.-n.-F. less salt.
Fresh	15.24	79.99	4.77	3.96	5.3
One month old	11.08	85.73	3.19	2.61	5.2

The diminution in solids-not-fat and salt show that the water had run out, and was not lost by evaporation. The salt has diminished in rather greater ratio than the water. This fact I have observed in butters kept in paper wrappers, and is possibly due to osmosis.

The results of the analysis of the fat are given in the following table :

ANALYTICAL RESULTS OF BUTTER FAT DURING 1894.

	French Butter.			English Butter.			New Zealand and Australian Butter.
Reichert-Wollny...	33.2 to 28.2	av. 30.6		32.3 to 24.1	av. 28.1		31.0 to 27.5 av. 29.1
Potash absorbed...	22.88 „ 22.49	„ 22.65		22.83 „ 22.01	„ 22.56		
Iodine	40.2 „ 31.3	„ 37.0		38.9 „ 37.1	„ 37.9		
Density $\frac{30.5^{\circ}}{30^{\circ}}$	„ 91165 „ 91123	„ 91143		„ 91174 „ 91036	„ 91116		Brittany Butter.
„ $\frac{30^{\circ}}{29^{\circ}}$	„ 90484 „ 90442	„ 90462		„ 90493 „ 90356	„ 90436		R.W. 28.8 to 28.6 av. 28.7
„ $\frac{100^{\circ}}{15^{\circ}}$				„ 8654 „ 8652	„ 8653		

THE RELATION BETWEEN SPECIFIC GRAVITY, FAT, AND SOLIDS-NOT-FAT IN MILK.

By H. DROOP RICHMOND.

IN the ANALYST (xix., 65) I proposed a new formula, $T = \cdot 2625 \frac{G}{D} + 1\cdot 2 F$, for calculating fat from total solids and specific gravity, and *vice-versa*. It was calculated from a considerable number of analyses made as accurately as possible, and using methods which seemed to me to be those most free from error.

Further series of analyses have shown that the results by this formula agree very much better with the actual results than those obtained by the milk scale. Evidence is not wanting that others have found that the fat calculated by the milk scale is slightly higher than that estimated. Thus Embrey (ANALYST, xviii., 120) states that he finds an average difference of 0·1 per cent., and proposes, and has even published, a set of tables corrected by this difference. More than one analyst has verbally informed me that in his laboratory the same thing has been noticed. In the "Proceedings of the Tenth Annual Convention of the Association of Official Agricultural Chemists," p. 108, a series of results was obtained showing that this new formula agrees better than any other, though, as it was not before them, it was not actually considered.

For these reasons I have calculated a table, by reference to which the total solids can be calculated from the specific gravity and the fat. This form of table is, I believe, the most useful now that rapid processes of fat-estimation are generally used, and the data used in a preliminary examination of milk are fat and specific gravity.

As the table is necessarily somewhat large, and it is frequently more convenient to calculate the results directly than to look them up in a table, I have endeavoured to simplify the formula without greatly detracting from the accuracy.

The formula $T = \cdot 2625 \frac{G}{D} + 1\cdot 2 F$ may be written :

$$T = \cdot 2625 \left(\frac{G}{1000 + \frac{G}{1000}} \right) + 1\cdot 2 F,$$

and, expanding this in terms of G , we get :

$$T = \cdot 2625 (G - \cdot 001 G^2 + \cdot 000001 G^3 \dots) + 1\cdot 2 F.$$

The first three terms only need be taken into account. This is equal to

$$T = \cdot 25 G + (\cdot 0125 G - \cdot 0002625 G^2 + \cdot 0000002625 G^3) + 1\cdot 2 F.$$

Now, for values of G likely to be obtained in practice—i.e., 20 to 36—the expression $\cdot 0125 G - \cdot 0002625 G^2 + \cdot 0000002625 G^3$ is nearly constant, and is equal to $0\cdot 14 \pm 0\cdot 02$.

Therefore the formula may be written :

$$T = \frac{G}{4} + \frac{6}{5} F + \cdot 14,$$

and the results will not differ (in extreme cases) more than ·02 from the other formula.

We find also that, if the fat is 3 per cent., the value of 0.05 F is 0.15, and is nearly equal to $0.125 G - 0.0002625 G^2 + 0.0000002625 G^3$, and the formula may be written :

$$T = 0.25 G + 1.25 F, \text{ or } T = \frac{G + 5 F}{4},$$

and this second simple formula is correct within 0.2 per cent. up to 6 per cent. fat. It is still more accurate if 0.05 per cent. be added for each 1 per cent. above 3 per cent., and subtracted for each 1 per cent. below 3 per cent.

A milk scale to express the same relation may be constructed on which 1 per cent. total solids = 1 inch, 1 per cent. fat = 1.2 inches, or 5 per cent. = 6 inches, and 1 degree of gravity = $\frac{1}{4}$ inch; if the zero on the fat scale be placed on a line with 5 per cent. on the total solid scale, the arrow will be in its correct position, 0.14 ($\frac{1}{7}$) inch below 20 degrees on the specific gravity scale.

It will be remembered that Hehnér and I gave as a simple formula $T = \frac{G}{4} + \frac{6}{5} F$; Babcock (*Ann. Report Wisconsin Exper. Stat.*, 1891, p. 298) has given the formula $T = \frac{G + 5 F}{4}$ in a slightly different form.

By placing an arrow 0.14 ($=\frac{1}{7}$) inch below the present arrow, existing milk scales will give a near approximation.

MAUMENÉ'S TEST FOR OILS.

By H. DROOP RICHMOND.

AFTER an extended trial of the various methods and forms of apparatus for performing the Maumené test which have been proposed from time to time, and after a study of the various reactions, so far as can be ascertained, which take place in that process, I have been led to devise the following method of procedure. By means of this modification, I feel assured that results more uniform and exact than those hitherto attainable can be secured :

Calorimeter.—A small, deep beaker is fitted inside a slightly larger one by means of a ring of cork; the outer beaker is placed in a tin cup padded with cotton-wool. The heat-capacity of this is estimated by placing 10 grammes of water inside, noting the temperature, and pouring in about 25 grammes of warm water (of known temperature), and noting the resulting temperature. The cooling in this calorimeter during a Maumené test does not amount to more than 1 per cent., and may be neglected.

Influence of Strength of Acid.—Between the limits, 92 per cent. to 100 per cent. H_2SO_4 , the rise of temperature is directly proportional to the strength of acid (difference not more than 1 per cent.).

Heat-Capacity of Mixture of Oil and Acid.—25 grammes of oil and 5 c.c. of sulphuric acid, when mixed, have a heat-capacity about equal to that of 20 grammes of water.

The total heat evolved per mean molecule may be calculated; this I call the "Relative Molecular Maumené" figure :

Let x = percentage of H_2SO_4 in acid (100 per cent. H_2SO_4 assumed as standard strength);

h = heat-capacity of calorimeter in grammes of water;

R = observed rise (25 grammes oil, 5 c.c. acid);

K = potash absorbed (19.5 per cent. KOH is assumed as a standard of comparison);

$$\text{then R.M.M.} = R \times \frac{21.5}{x - 78.5} \times \frac{20 + h}{20} \times \frac{19.5}{K}$$

This action of the acid on the oil is probably in two stages—(i.) hydrolysis, (ii.) action on the unsaturated bonds.

I believe that a definite amount of the heat evolved is due to hydrolysis, and the remainder to the second stage. My experiments indicate that the R.M.M. of hydrolysis lies between 7° and 18° , and is probably about 12° , and that glycerides containing acid radicles with two unsaturated bonds evolve more than twice the amount of heat than those with one unsaturated bond.

Contribution to Butter Analysis—The Abbé-Zeiss Refractometer. J. Delaite. (*Bull. de l'Assoc. belge des Chimistes*, 1894, No. 5, pp. 145-152.)—According to Zeiss, the refractive index may be taken at any temperature, and the reading subsequently calculated to a standard temperature, *e.g.*, 25° . But the author proves that this does not give concordant results with fats. Thus, the indices obtained from one butter, from readings taken at different temperatures and reduced to $25^\circ C.$, were:

Temp. of Observation.	Refractive Index, calculated to $25^\circ C.$
45°	48.8
40°	49.3
35°	49.5
30°	49.65
25°	49.7

This inverse ratio of the refractive index to the temperature only occurs markedly in the case of pure normal butters. Abnormal and adulterated butters and margarines, though not giving constant results, show no order in their variation. Two margarines, for instance, gave the following results:

Temperatures.						Indices calculated to $25^\circ C.$				
	45°	40°	35°	30°	25°	45°	40°	35°	30°	25°
Ref. Indices (1) —	49.6	52.2	55.3	—	—	—	58	57.8	58.1	—
„ (2) 47	49.5	52.2	55	59.2	—	58.2	57.9	57.8	57.8	59.2

It is necessary, therefore, to select a definite temperature for taking the reading, and it is suggested that this should be $40^\circ C.$ for fats and 25° for oils. By means of a current of warm water the temperature is brought to 45° or 50° , and is then allowed to fall very slowly to 40° . The error of reading introduced by the temperature of the whole instrument not being the same as that of the water, is thus avoided.

As to the value of the process, the author's opinion is that it cannot be relied upon to give alone even a preliminary decision as to the genuineness or otherwise of a butter. For although the average butter does not give a refractive index higher

than 44 at 40°, yet samples undoubtedly genuine often give higher figures. On the other hand, abnormal and adulterated butters have been known to give figures as low as 41.6 and 42. Moreover, rancidity appears to affect the result. A butter known to be adulterated was left for more than a year, and, when strongly rancid, gave a refractive index of 48.25 at 30°, while that of an average pure butter at that temperature is 52.5. Hence, judged by the refractive index alone, this sample would have been declared genuine. The addition of cocoanut oil or palm oil to a butter also lowers the index. The refractive index of the former varies from 33.2 to 35.2 at 40°, that of the latter from 35.2 to 45.3. A mixture of a pure butter with refractive index of 44 at 40°, with 20 per cent. of cocoanut oil, showed an index of 42.2. A similar result was also obtained by adding horse-fat to butter. C. A. M.

An Examination of the Twitchell Method for the Determination of Rosin in Soap. T. Evans and I. E. Black. (*Amer. Chem. Journ.*, 1895, xvii., I., pp. 59-67.)—Twitchell's process,* although the most satisfactory known, has been proved to be far from exact, especially when the gravimetric method is employed, in which, as has been shown by Lewkowitch, a loss of from 10 to 15 per cent. is frequent.† With the object of rendering it more accurate, the authors have made experiments on the same lines as Twitchell, preparing test-samples containing known quantities of rosin and fatty acids.

In the volumetric method, too high results were obtained when the combining weight of the rosin was taken as 346, the number given by Twitchell. At first this was attributed to incomplete etherification, but repeated titration proved that the combining weight of the sample was 328.9—a figure lower than any of those recorded by Twitchell or Lewkowitch. Taking this number as the factor, the results were much more accurate, as is shown in the following instances selected from a long table :

PERCENTAGE OF ROSIN.

Theory.	Combining Weight 346. Found.	Difference.	Combining Weight 328.9. Found.	Difference.
40.13	42.19	+ 2.06	40.10	- 0.03
40.13	41.74	+ 1.61	39.67	- 0.46
40.13	42.16	+ 2.03	40.06	- 0.07

By leaving the alcohol saturated with HCl, and before diluting with water, all night on the water-bath, the results could be considerably lowered, and still more so by adding H₂SO₄ before this treatment. The substitution of methyl alcohol for ethyl alcohol gave poorer results, and if left all night before evaporating the alcohol caused considerable loss. The results were also too low if the water surrounding the flask during etherification became too warm, or if the current of HCl were too rapid.

By using ethyl alcohol, and boiling out soon after etherization, concordant results were obtained, approximating closely to the theoretical.

Attempts were made to etherize rosin alone, but in each case the results were too low.

* See ANALYST, xvi., p. 169.

† *Journ. Soc. Chem. Ind.*, xx., 1893.

Weight of Rosin.	Rosin calculated from Titration.	Difference.	Percentage loss.
2.0968 grms.	2.054	0.0428	2.00
2.4723 "	2.45	0.0223	0.90
2.035 "	2.016	0.019	0.93
2.03 "	2.016	0.014	0.69
2.115 "	2.091	0.024	1.14

With methyl alcohol the loss was still greater, being 6 or 7 per cent., a result probably due to decomposition.

As is shown in the first table, when the combining weight of a rosin is known the volumetric method is sufficiently accurate, but the combining weight varies greatly in different grades of rosins, ranging from 328.9 to 355.9. Accordingly, the authors made the attempt to separate the rosin in a given sample, determine its combining weight, and to use this number as the factor in the volumetric determination. A sample of resin (combining weight 328.9) was subjected to the gravimetric process, and the rosin acids dried at 105°, and titrated. The combining weights obtained were (1) 328.6, (2) 325.9.

Four similar experiments with a rosin with combining weight 351.4 gave the combining weights, 329, 323.9, 331.1, 329.4.

The explanation of these discrepancies was looked for in the loss which occurs in the gravimetric method. This loss was found to be in the petroleum ether layer from which the saponifiable rosin had been removed by caustic potash. A sample of rosin (combining weight 351.4) was treated as in the gravimetric method, and the residual petroleum ether, after removal of the rosin, evaporated to dryness, when a gum-like mass with a turpentine smell was left, corresponding to 9.71 per cent. of the original rosin. This residue was not saponifiable with alcoholic potash, and was therefore not an ether.

A mixture of fatty acids and rosin gave a loss of 5 per cent., which corresponded exactly with the percentage of unsaponifiable matter in the sample of rosin used.

Four other samples of rosin tested for unsaponifiable matter gave the following results :

Grade.	Unsaapon. Matter per Cent.	Comb. Weight Original Rosin.	Calculated Combining Weight Saponifiable Rosin.
WW	7.34	339.9	315.0
WG	5.00	328.9	312.4
N	9.00	351.4	319.7
N	8.21	347.5	319.0
M	7.61	339.8	314.0

Mean 316.0

The authors are investigating the subject further, and perfecting a method for the separation of the unsaponifiable rosin from the hydrocarbon oils used as soap-fillers, etc.

C. A. M.

The Determination of Sulphur in Volatile Organic Compounds. C. F. Maybury. (*Amer. Chem. Journ.*, 1894, xvi. 544; through *Chem. Zeit.*)—The author has made a critical examination of various methods of determining sulphur, especially

in petroleum products, and has in particular examined the method of Carius and the process consisting in oxidizing the material to be analysed in a combustion tube in a stream of air or oxygen. The tube, which must be of refractory glass, is somewhat contracted in the middle; the stream of air or oxygen is let in through a piece of narrow tubing passing up to the point of contraction. The stopper which carries this narrow tube has a second perforation carrying a tube conveying carbonic acid, in order to drive the material to be analysed towards the forward end of the combustion tube. The products of combustion pass into a large U-tube containing 50 c.c. of caustic potash, of which each c.c. is equivalent to 1—5 milligrammes of sulphur, according to the content of the substance to be analysed. The liquid can be titrated in the U-tube itself, methyl orange being used as an indicator.

B. B.

The Determination of Free Sulphuric Acid in Leather. P. F. Jean. (*Rev. Chim. Analyt.*, 1895, iii. 13; through *Chem. Zeit.*)—The process preferred by the author consists in extracting the dried and powdered leather in a Soxhlet apparatus with absolute alcohol containing a little caustic potash to fix the sulphuric acid. After thorough extraction, the alcohol is distilled off and the sulphuric acid determined in the residue as barium sulphate in the usual manner.

B. B.

Thioacetic Acid as a Substitute for Sulphuretted Hydrogen in Analysis. R. Schiff and N. Tarugi. (*Gazz. Chim. ital.*, 1894, xxiv, vol. ii, 551; through *Chem. Zeit.*)—When a hydrochloric acid solution of the metals of the second group is treated with a slightly ammoniacal solution of ammonium thioacetate (30 per cent. strength) in the proportion of 1·5—2 c.c. for 1 gramme of the metals to be separated, and the mixture is warmed to 80–90° C., all the metals are precipitated as sulphides, although only a slight smell of sulphuretted hydrogen is perceptible. After cooling and filtering, the solution is found to be free from metals of the second group, and the analysis is completed in the ordinary manner.

Ammonium thioacetate is decomposed in the presence of hydrochloric acid with the formation of ammonium chloride, acetic acid, and sulphuretted hydrogen. Tin, bismuth, copper, platinum, and gold are only partly precipitated in the cold, but completely on keeping. Lead and mercury are precipitated in the cold as red chlor-sulphides, which on heating are converted into the ordinary black sulphides. With arseniates the reagent gives a slight white turbidity in the cold, but on heating the solution sudden and complete precipitation takes place. Cadmium sulphide dissolves in the warm hydrochloric acid, but again separates on cooling. Thioacetic acid reduces ferric salts and chromates.

The reagent is prepared by adding 10 per cent. ammonia solution in slight excess to thioacetic acid, and then diluting the mixture to treble the volume of the thioacetic acid used. The resulting liquid is yellow, and smells slightly of ammonium sulphide; it becomes turbid on heating, and needs to be freshly prepared every eight or ten days. The advantages which its use presents consist in avoidance of the smell of sulphuretted hydrogen, and of the need for a Kipp or similar apparatus; the rapidity and completeness of precipitation are also reasons for preferring this reagent.

B. B.

The Phospho-molybdic Acid Test as applied to Lard Analysis. George F. Tennille. (*Jour. Amer. Chem. Soc.*, 1895, xvii. 1, pp. 33-41.)—The conclusion arrived at by Samelson (*ANALYST*, xix., p. 251), that the phospho-molybdate test is of little analytical value, is here confirmed. It will certainly detect the addition of a large quantity of cotton-seed-oil; but if only a slight green coloration be obtained, adulteration cannot be considered as proved.

The author's experiments were made with the three grades of lard constituting the bulk of that sold to the New York refiners—viz.:

No. 1 Lard.—Steam-rendered, and consisting of gut fat, with occasionally a little trimmings, but no leaf.

Prime City Lard.—Rendered either by steam or in open kettles, and consisting of trimmings, head, foot, and back fat, and sometimes a little leaf.

Prime Western Lard.—Steam-rendered; from gut fat, with occasionally trimmings.

All the samples of No. 1 lard examined gave the green coloration, and this was also the case with a sample rendered in the laboratory from No. 1 stock. This was No. 10 in the table.

Nearly all the City lards gave negative results. Of fifty-seven undoubtedly pure samples, fifty gave no coloration, and seven gave a slight green tinge.

The Western steam lard often gave a slight green colour, which, however, was not so pronounced as that obtained with No. 1 lard.

The following table gives the particulars of the tests applied to ten samples of No. 1 lard:

Hübl Figure.	Dalican's Titre °C.	Rise of Temperature with H_2SO_4 °C.	Becchi's Silver Test.	Milliau's Silver Test.	Phospho-Molybdic Acid Test.	Crystals from Ether Powder. Micro-scope.	Free Fatty Acid, per Cent.	Spec. Grav. 98° C: 15° C.
1. 54.5	41.4	30.5	Negative.	Slight discoloration.	Decided green. Blue if alkaline.	Lard only.		
2. 55.6	41.4	31.8	„					
3. 58.6	40.2	31.8	„	„	„	„	0.73	0.8595
4. 53.7	41.4	30.0	„					
5. 55.4	41.4	31.5	Slight discoloration.					
6. 56.1	41.4	31.7	Negative.					
7. 47.7	42.4	25.5	Slight discoloration.					
8. 51.7	41.8	31.5	Negative.	„	„	Indication of beef.		
9. 52.0	41.9	29.5	Slight discoloration.	„	„	„		
10. 48.0	—	—	—	„	„	—		

As leaf lard does not give a green colour with the reagent, the author thinks that the favourable reception given to the test on its first appearance was due to the experimenters using that lard.

G. A. M.

The Asphalt Question. S. F. Peckham. (*Jour. Amer. Chem. Soc.*, 1895, xvii., No. 1., pp. 55-63.)—In a critical review of various papers that have been pub-

lished on the chemistry and analysis of asphalt, the author states his objection to the method employed by Richardson of determining the amount soluble in CS_2 , since that liquid does not dissolve all the bitumen. With Miss Linton's process,* in which the use of CS_2 is altogether discarded, he obtained very concordant results, agreeing to the third place of decimals. The petroleum ether used should be obtained from petroleum consisting of hydro-carbons of the paraffin series, and have a specific gravity of 0.7. D. Torrey's method, based on successive solutions of the asphalt in alcohol, does not offer any advantages over that proposed by Miss Linton.

C. A. M.

Rapid Estimation of Starch. P. L. Hibbard. (*Jour. Amer. Chem. Soc.*, 1895, xvii. 1, pp. 64-68.)—Of the methods for determining starch in crude celluloses, none can claim absolute exactness. Lintner's process of heating in closed vessels, under high pressure, with water or a weak acid, gives good results when soluble convertible carbo-hydrates other than starch are absent; while Märker's process, which is the most satisfactory, has the same drawback, more or less of the various gums present being dissolved by the diastase. In the latter process the substance is boiled for a few minutes with water to gelatinize the starch, and after cooling to 60° diastase is added, and the liquid maintained at 65° for an hour. The solution is then filtered, the filtrate treated with HCl , and the converted dextrose estimated by Fehling's solution (see ANALYST, xx., pp. 17-19).

A shorter and easier modification of this method is suggested by the author, the advantages of which over older processes are: (1) Prevention of clots by the addition of malt extract previous to heating; (2) prevention of lactic fermentation by rapid heating; (3) a minimum solution of bodies other than starch, owing to the short time of boiling.

The malt extract which is used instead of pure diastase is prepared by infusing the malt for several hours, with water containing 15 to 20 per cent. of alcohol, and filtering. The alcohol acts as a preservative, without interfering to any great extent with the diastatic power of the malt. The solution thus prepared will keep for a fortnight, even in warm weather.

In the estimation the finely-powdered substance is heated to boiling in a flask, together with 50 c.c. of water and one to two c.c. of malt infusion. (If the substance contain much fibrous material and but little starch, nothing is gained by adding the malt here, as the diastase is destroyed by the high temperature before it can act on the starch.) After boiling for a few minutes, the mixture is cooled to 50° or 60°C. , and another two or three c.c. of malt extract added. It is then slowly heated for ten to fifteen minutes till boiling, again partially cooled, and tested with iodine solution. If any blue colour appear, the treatment with malt must be repeated. When all the starch is changed the liquid is cooled, made up to definite volume, and filtered through muslin or linen. An aliquot part, sufficient to contain 0.2 to 0.3 grammes of starch, is taken from the filtrate, and is boiled on a sand-bath, in a 100 c.c. flask, with 5 c.c. of HCl (30 per cent. gas), and sufficient water to make up about 60 c.c. No reflux

* ANALYST, xx., p. 41.

condenser need be used, and the conversion is complete after thirty minutes' actual boiling. When cool, the solution is neutralized with NaOH, and the dextrose estimated with Fehling's solution. Less than two hours is required for the determination. The following figures compare the results obtained by this method with those by the direct inversion method :

Material.			Moisture.		Starch per Cent.	
					Malt Method.	Acid direct.
Starch factory feed	10.0	...	10.0	40.9
Impure moist starch	12.0	...	81.3	82.3
"	"	...	—	...	79.5	80.9
Good starch	12.8	...	84.3	84.6
Purified dried starch	—	...	95.1	96.5
Corn, large white kernels	11.0	...	63.5	68.2
Wheat, hard red variety	10.0	...	60.5	65.0
Bran from same wheat	11.3	...	28.4	44.4
Middlings from same wheat	10.1	...	53.8	60.0
Low flour	"	"	10.2	...	66.0	67.7
Good flour	"	"	11.0	...	67.9	69.0
Wheat flour	10.0	...	70.7	73.6
Wheat bran	10.1	...	29.3	46.5

C. A. M.

A New Method of Estimating Carbon in Iron. Leop. Schneider. (*Stahl und Eisen*, 1894, 1029; through *Chem. Cent. Blatt.*)—The author has found that the addition of powdered copper and lead to the iron causes the latter to burn much more readily in a current of oxygen. The lead is prepared by shaking the molten metal, and the copper by reduction of oxidized copper-wire in a current of hydrogen. In the estimation 3 grammes of the finely-divided steel are incorporated with 10 grammes of a mixture of three parts of lead-powder with one part of copper-powder, and introduced into the combustion-tube in a porcelain boat, below which is placed a little asbestos. In front of the boat is placed 10 to 20 cm. of copper oxide. After the boat has been brought to a dull red heat, the current of oxygen is admitted, and the metallic mixture readily takes fire. The gases produced, after being dried by H_2SO_4 , are led into soda. After the combustion the current of oxygen is continued for another ten minutes. The whole operation requires three-quarters of an hour.

C. A. M.

On the Use of Litmus and Methyl Orange as Indicators in Volumetric Analysis. G. Lunge. (*Zeit. für angewandte Chemie*, 1894, 24, pp. 733-738.)—The author admits that litmus, when prepared and used according to Reinitzer's directions (*ANALYST*, xix. 255), is more sensitive than methyl orange, but denies that the superiority is as great as claimed. With 250 c.c. of distilled water, and using $\frac{1}{8}$ normal or decinormal acid, it is not eight times, but only twice, as sensitive. Since Reinitzer only gave comparative results when the indicators were used with distilled water, the author has made experiments to determine their merits when used with 50 c.c. of a $\frac{1}{8}$ normal soda solution. With normal acid practically identical results are obtained, but methyl orange is preferable on account of its speed and the

precautions to be observed in the use of litmus. With semi-normal acid the change of colour is more difficult to observe in the case of methyl orange, but a practised observer can be sure to a drop; and when liquids containing carbonic acid have to be frequently titrated, the gain of time is a compensating advantage. It is only with decinormal acid that litmus is undoubtedly superior, and Reinitzer's method of titration must be observed.

C. A. M.

The Determination of Potash in Manures. W. E. Garrigues. (*Jour. Amer. Chem. Soc.*, 1895, xvii., No. 1, pp. 47-51.)—The process recommended depends on (1) conversion of all the potash present and removal of ammonia and organic matter by ignition with H_2SO_4 ; (2) precipitation of the H_2SO_4 with BaCl_2 ; (3) precipitation of the excess of barium and other earthy bases with Na_2CO_3 ; (4) evaporation of the filtrate with PtCl_4 .

In the estimation, 10 grammes of the sample are ignited in a platinum basin with concentrated H_2SO_4 , finishing with a blast. The residue is powdered in a mortar with a little hot water, washed into a 500 c.c. flask, and boiled for half an hour with 250 c.c. of water. A 10 per cent. solution of BaCl_2 is then introduced, 5 c.c. at a time, until no more precipitate forms. One drop of phenol-phthalein solution is then added, and sodium carbonate until a faint pink tinge is observed. The liquid is boiled, cooled, made up to the mark, and filtered. To 50 c.c. of the filtrate (= 1 gramme of the sample) HCl is added to slight acid reaction, then PtCl_4 , and the liquid evaporated to dryness on the water-bath. The precipitate is washed with alcohol (specific gravity 0.848), dried at 100° , and weighed. For potassium salts ignition with H_2SO_4 is omitted.

The author lays stress on the following points: (1) Cautious addition of the H_2SO_4 ; (2) fine pulverization of the ignited mass in order to prevent calcium sulphate mechanically enclosing potassium sulphate; (3) avoidance of an excess of Na_2CO_3 , which might cause decomposition of the precipitated BaSO_4 .

From the table of results given, this method, which, in common with other methods, does not account for the entire potash present in samples of known composition, appears to be superior to the Lindo-Gladding process.

C. A. M.

The Addition of Calcium Chloride to the Solution of a Fertilizer in the Determination of Potash. Rudolf de Roode. (*Jour. Amer. Chem. Soc.*, 1895, xvii., No. 1, p. 46.)—In order to allow platinum basins to be used in the Lindo-Gladding method, Huston recommended the addition of sufficient CaCl_2 to form calcium phosphate with the phosphoric acid present. The author finds that, in order to make this effective, it is necessary to filter off the precipitate formed by the CaCl_2 and NH_3 , and then to add ammonium oxalate to the filtrate. As this involves two filtrations, the suggestion does not seem to be an improvement on the present alternative method.

C. A. M.

Viscometrical Examination of Butter. C. Killing. (*Zeit. für angewandte Chemie*, 1894, pp. 643-645, and pp. 739, 740.)—The apparatus used by the author

consists of a wide glass cylinder, closed at the bottom by an indiarubber cork, through which passes a short tube, having its top ground to receive a sort of pipette. This holds about 50 c.c., and has an arrangement in its body to allow of the introduction of a small thermometer. The upper tube of the pipette, which passes through the top cork of the cylinder, is closed by a tap, and the pipette has three marks—one below the body and two above, the latter being placed about 1 cm. apart. The cork closing the top of the cylinder is made to remove in two halves, and a second thermometer is passed through one of these. The whole apparatus is fixed in a clamp, and a beaker is placed below to receive the fat.

The standard of viscosity is the time a definite volume of water at 20° C. takes to run out. The viscosity of butter is taken at 40° C., and it is essential that both thermometers should be compared with a standard thermometer, and a correction made if different.

In using the apparatus the top cork of the cylinder, with the thermometer, is removed and the pipette withdrawn. About 60 c.c. of the clear melted fat is cooled to 40·5°, the pipette thermometer inserted in its place, and the butter sucked to the upper of the two top marks. The tap is then closed, and the pipette placed in the cylinder; the latter is filled to the top with water at 42°, and the cork covers replaced. When both thermometers stand at 40°, the tap is slightly opened and the fat allowed to run slowly down to the lower of the upper marks. The tap is then opened wide, and the fat runs out into the beaker until it reaches the lowest mark, when the tap is closed and the time noted. The mean of two or three determinations, which should not vary more than $\frac{1}{4}$ to $\frac{1}{2}$ a second, is taken, and this compared with water as 100, gives the viscosity number.

In the author's apparatus, distilled water at 20° C. ran out in 80·33 seconds, and supposing a butter to take 3 minutes 42 seconds, this would give as the viscosity number

$$\frac{222\cdot00 \times 100}{80\cdot33} = 276\cdot3.$$

The following tables give the results obtained with different samples of margarines and butters:

A.—MARGARINES.

Description.		Time running out.		Viscosity Number.	Remarks.
		Min.	Sec.		
1.	Dutch margarine	4	12·50	314·3	From the same factory. (All from factory. No. 4 contained earth-nut oil; Nos. 5 & 6 cotton-seed & sesame oil.
2.	German margarine, M.D.	4	13·66	315·8	
3.	„ „	4	11·66	313·3	
4.	„ „ M.B.	4	11·66	313·3	
5.	„ „	4	13·83	315·9	
6.	„ „	4	11·83	313·5	
7.	Rotterdam margarine	4	12·16	313·9	From same factory.
8.	German margarine, M.G.	4	12·38	314·2	
9.	„ „	4	15·00	317·4	
10.	„ „ M.H.	4	14·00	316·2	
11.	„ „	4	11·83	313·5	
Mean		4	12·77	314·7	

B.—BUTTERS.

Description.		Time running out.	Viscosity Number.
1. Westphalian	...	3 min. 42.00 sec.	276.3
2. Rhine	...	3 .. 41.90 ..	276.2
3. "	...	3 45.33	280.5
4. Dutch	...	3 42.16	276.5
5. Oldenburg	...	3 45.16	280.3
6. Westphalian	...	3 43.42	278.1
7. Holstein	...	3 46.00	281.3
8. Rhine	...	3 44.13	279.0
Mean	...	3 min. 43.76 sec.	278.5

Taking the mean numbers, the amount of margarine in an adulterated butter may be calculated from the formula :

$$x = (v - 278.5) \frac{100}{314.7 - 278.5}, \text{ or } x = 2.76(v - 278.5), \text{ where } v = \text{viscosity number.}$$

The products used in the manufacture of margarine gave the following viscosity numbers :

German oleo-margarine	339.2
American	332.7
Earth-nut oil	296.3
Sesame oil	273.9
Cotton-seed oil	258.9

A mixture of 75.6 parts of American oleo-margarine with 24.4 parts of cotton-seed oil gave the mean viscosity number of margarine 314.7. A mixture of 50 per cent. of each gave a viscosity number of 295.8, but the product was so fluid that it would have been unsaleable. It thus appears that it is impossible to make a butter from cotton-seed oil and oleo-margarine with the right viscosity number—viz., 278.5.

The one oil from which an artificial butter with a low viscosity number can be prepared is cocoa-nut oil, which gives 223.1, but according to the author this is no longer employed in the manufacture of margarine, since it has been found impossible to get rid of the taste.

Mixtures of butter and margarine with the respective viscosity numbers of 278.8 and 315.6 showed a proportionate increase in the numbers :

Butter Fat.		Margarine Fat.	Viscosity Number Found.	Corresponding to Margarine %.
95.17	+	4.83	280.20	3.81
75.53	+	24.47	287.56	23.83
55.70	+	44.30	295.50	45.42
42.45	+	57.55	300.16	58.09
25.20	+	74.80	306.09	74.23

The smallest amount of added margarine that can be detected will depend to a large extent on the amount of variation of butters among themselves, and this seems to be considerable. Butter from the milk of a cow fed on beetroot only gave a viscosity number of 270.76, whilst another cow fed exclusively on distillers' refuse produced a butter with viscosity number 278.23. Nevertheless, the author claims for this process superiority over the Reichert-Meissl method.

C. A. M.

The Rapid and Accurate Analysis of Bone-black. W. D. Horne. (*Journ. Amer. Chem. Soc.*, 1895, xvii., 1, pp. 51-55.)—In new char the amount of moisture is of importance, being frequently limited by contract to 3 per cent. It is determined by heating 2 grammes for two hours in an air-bath at 140° C.

Calcium carbonate may be conveniently estimated in Lunge's nitrometer, using mercury. The flask containing the sample and the small tube of HCl is connected with the three-way cock of the nitrometer, and the liberated CO_2 measured with the usual corrections for temperature and pressure. The char in the flask is previously moistened with 3 c.c. of saturated solution of mercuric chloride, which absorbs any H_2S formed.

The carbon, iron, calcium sulphate, and insoluble ash are determined on one portion. This is prepared by first removing all particles of iron with a magnet, and then grinding to an almost impalpable powder. Two grammes of this are boiled in a beaker with 20 c.c. HCl for thirty minutes, and the beaker then filled up with boiling water. The residue settling down is washed several times by decantation, and then transferred to a weighed platinum Gooch's crucible with asbestos plug. It is then washed with 80 per cent. alcohol, followed by 90 per cent. alcohol, and finally with ether, after which it is dried in the oven at 100° and weighed. This gives the carbon and the ash, and the loss of weight on burning over a blast when deducted from this gives the carbon alone.

The acid filtrate is nearly neutralized with ammonia, and ammonium acetate added till the solution turns yellow, then a few drops in excess, and the liquid heated below 70° C. until the iron and aluminium phosphates are completely precipitated. The precipitates washed free from chlorides are dissolved in a 6-oz. flask by dilute H_2SO_4 , 10 c.c. of sodium sulphite added and the liquid boiled until the iron is completely reduced and the SO_2 expelled, after which it is cooled and titrated with permanganate. If the aluminum is to be determined, the above precipitate may be treated with 150 c.c. of ammonium molybdate solution and filtered from the phosphoric acid. The iron and aluminum in the filtrate are then precipitated by ammonia, the precipitate filtered off, dissolved in HCl, and reprecipitated by ammonia, collected, ignited, and weighed. The two may then be separated by any of the known methods.

The filtrate from the iron and alumina precipitation which contains the CaSO_4 is acidified with HCl, the sulphuric acid precipitated by BaCl_2 , and the BaSO_4 calculated to CaSO_4 .

Calcium sulphide is estimated by evaporating 5 grammes of char nearly to dryness with 20 c.c. HNO_3 , and after adding 20 c.c. of HCl again evaporating low to expel the HNO_3 . The residue is taken up in HCl, and the sulphuric acid precipitated in an aliquot part of the filtered solution. From the BaSO_4 is deducted that found above as CaSO_4 .

The physical conditions of the char should be thoroughly examined. The grist is estimated by shaking gently 100 grammes on sieves of known mesh and weighing what passes through.

The density is determined, both loose and packed, by introducing the char into a weighed 50 c.c. or 100 c.c. flask, making up to the mark, and weighing. From this the density and the weight per cubic foot is calculated.

The porosity is determined by introducing the char little by little into the weighed flask filled with distilled water. The flask is tapped to pack the char, and when filled up to the mark is heated on the water-bath to expel air. After cooling, the supernatant water is removed, and the flask and its contents weighed. The increase of weight over that of the flask packed with char gives the amount of contained water.

To determine the durability of a char under continued friction, the author has devised the following test: 25 grammes of the char are shaken ten times backwards and forwards in a sieve with circular holes one-fiftieth of an inch in diameter, and the dust which passes through weighed. Dust and char are then shaken together in a cylinder of turned iron two inches deep and four inches in diameter with six glazed porcelain marbles. After shaking 200 times the marbles are removed, the char sifted as before, and the increased weight of dust calculated to per cent. of the char used. Fairly constant results were obtained from different parts of the same sample. Thus a new char yielded 1.72, 1.46, and 1.76 per cent. of dust. Two grades made by one firm gave 2.16 and 2.16 per cent. in one case, and 2.86 and 2.87 per cent. in the other. Char which had been in use ten months gave 0.92 and 0.94 per cent., which showed that its wearing condition had improved by use, the softer parts having been separated.

C. A. M.

On the Examination of Linseed Oil and Boiled Linseed Oil. Dr. Hugo Amsel. (*Zeit. für angewand. Chemie*, 1895, Heft. iii., pp. 73-78.)—With the exception of the Köttstorfer number, none of the usual tests applied to an oil (e.g., the specific gravity, iodine-number, elaidin reaction, etc.), is of much value in the case of linseed oil, since they give results varying so widely in different specimens of the pure oil.

The method proposed by Filsinger (*Chem. Zeit.*, 1894, 1005 and 1867), in which the sample is mixed with chloroform and alcohol, and examined with the polariscope, is useless when mineral oil, which is optically inactive, has been used to adulterate the linseed oil. Moreover, in the case of boiled oils the solution is often so dark as to prevent an accurate observation being made.

As the result of an experience of many years, the author declares that adulteration may be detected with certainty by determining:

1. The capacity of the oil to dry when smeared on glass.
2. The saponification number, and the behaviour of the alcoholic solution on adding water.

And, according to the results from the above,

3. The mineral matter.
4. The amount soluble in alcohol.

Pure linseed oil without "driers" ought to dry quite hard in three days at most when spread in a thin layer on glass. With the addition of 5 per cent. *driers* it should not require longer than twenty-four hours, which should also be the time taken by boiled linseed oil. The addition of only 5 per cent. of rape oil prevents the layer from becoming hard, even after eight days.

In testing an oil the Köttstorfer number is first taken, about two grammes being saponified in the usual way with alcoholic potash. Since mineral oils are not thus saponifiable, and the saponification number of rosin oils very low, the result will give

an approximate idea as to the character of the oil. The average saponification number (*i.e.*, the KHO required per 1000 of oil) of linseed oil is 188, while that for boiled linseed oil varies from 190 to 195.

The drying test is next made, and this will prove whether mineral oils be present. If present, the thin layer on the glass will not be hard after three days.

Another important characteristic of a pure linseed oil is that its alcoholic soap solution remains perfectly clear on adding water, whilst an oil adulterated with mineral oil, rosin, or resinated metallic oxides becomes milky. The following instances, selected from a long table, show this :

TABLE I.

	Saponifica- tion No.	Alcoholic Soap Solution + Water.	Calculated Addition from the Saponification No. per cent.
1. Linseed oil	188	Clear	
2. Boiled linseed oil	196	"	
3. Blue rosin oil	13.5	Milky	
4. American mineral oil	2	"	
7. Scotch mineral oil	9.6	"	
10. Linseed oil + 5 per cent. mineral oil	177	"	5.9
11. Linseed oil + 10 per cent. mineral oil	171.4	"	9
16. Linseed oil + 20 per cent. rosin oil	156	"	18
22. Boiled linseed oil + about 2.5 per cent. resinated metal- lic oxides	187	"	About 3

The saponification number of No. 22 was so nearly normal that had the alcoholic soap solution remained clear it would readily have been passed as genuine.

TABLE II.—LINSEED OILS.

	Saponification No.	Alcoholic Soap Solution on addition of Water.	Iodine No.	Drying Capacity on Glass + 5 per cent Driers.
1.	183	Clear	180	Hard in 24 hours.
2.	188	"	180	
3.	173	Milky	—	Viscous after 24 hours.
4.	179	"	112	After 5 days, as at beginning.
5.	189	Clear	180	Hard in 24 hours.
6.	180			
7.	174	Milky		Viscous after 24 hours.
8.	165		157	Viscous after 48 hours.
9.	190	Clear	183.5	Hard in 24 hours.
10.	191		167	After 5 days, as at beginning.
11.	190			Hard in 24 hours.
12.	189			Hard in 24 hours.

The foregoing tests showed that Nos. 1, 2, 5, 6, 9, 11, and 12 were pure linseed oils. Nos. 3 and 7 were adulterated with rosin oil; No. 8. with mineral oil; and Nos. 4 and 10 with rape oil.

TABLE III.—BOILED LINSEED OIL.

	Saponifica- No.	Alcoholic Soap Solution + Water.	Iodine No.	Drying Capacity.	Soluble in Alcohol, per cent.	Mineral Matter, per cent.
1.	117	Milky	—	After 3 days viscous		
2.	141	"	153	Dry after 2 days.		
3.	188	"	—	"		
4.	193	Clear	—	Hard in 24 hours.		
5.	138	Milky	128	Not quite hard after 2 days		
6.	153	"	—	Dry after 2 days		
7.	195	Clear	181	Hard in 24 hours		
8.	172	Milky	—	Dry after 2 days	26.3	
9.	199	Clear	174	Hard in 24 hours		
10.	92	Milky	148	Dry in 36 hours	50	
11.	198	Clear	183	Hard in 24 hours.		
12.	191	"	—	"	24	
13.	197	"	189	Hard in 24 hours.		
14.	189	Milky	165	Dry after 2 days.	32	
15.	193	Clear	169	Hard after 24 hours.	19.5	
16.	174	Milky	155	Still viscous after 3 days	19	0.21
17.	171.8	"	146	"	18	0.21
18.	175	"	161	Dry after 2 days	25	0.23
19.	191	Clear	125	After 7 days as at first	8	
20.	125	Milky	—	Dry after 2 days	37.8	0.4
21.	190	Clear	—	Hard in 24 hours		
22.	193	"	—	Hard in 18 hours		
23.	161.7	Milky	—	Viscous after 2 days		
24.	133	"	—	Dry after 2 days		
25.	183	"	—	"		

Of the above, Nos. 4, 7, 9, 11, 12, 13, 15, 21, and 22 were pure boiled linseed oil. No. 19 contained 50 to 60 per cent. of rape oil. Nos. 16 and 17 contained mineral oil. Nos. 1, 2, 3, 5, 6, 8, 10, 14, 18, 20, 23, 24, and 25 contained rosin oil, or resinated metallic oxides.

TABLE IV.—LINSEED OILS, THICKENED BY HEAT AND OXIDATION.

	Saponification No.	Alcoholic Soap Solution + Water.	Iodine No.	Per cent. Soluble in 98 per cent. Alcohol.
1.	195	Clear	94.4	18.5
2.	194	"	104	17.7
3.	209	"	—	9.2
4.	192	"	—	6
5.	111	Turbid	—	49

Of these, No. 5 was adulterated with about 40 per cent. of rosin, while the others were pure linseed oils.

In the case of thickened oils, the determination of the amount soluble in alcohol is of value. About 1 gramme of the oil is mixed with 20 c.c. of alcohol, allowed to stand at the ordinary temperature for twenty-four hours, with constant shaking, and then filtered. For linseed oils the mean amount is about 25 per cent., while thickened oils always yield under 20 per cent.

C. A. M.

THE ANALYST.

APRIL, 1895.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held the evening of Wednesday, March 6, at the Chemical Society's Rooms, Burlington House, the President (Dr. Stevenson) occupying the chair.

The minutes of the previous meeting were read and confirmed.

Mr. D. J. O'Mahony, Public Analyst for Cork, was elected a member of the Society.

Dr. Dupré read the following paper :

NOTE ON THE CHEMICAL AND BACTERIOLOGICAL EXAMINATION OF WATER, WITH REMARKS ON THE FEVER EPIDEMIC AT WORTHING IN 1893.

By A. DUPRÉ, Ph.D., F.R.S., F.I.C., Lecturer on Chemistry at the Westminster Hospital Medical School.

It has long been known that certain diseases, as cholera or typhoid fever, were liable to be spread by water which had been contaminated by discharges coming from persons suffering from these disorders. It is, however, only within comparatively speaking recent years that this liability of spreading disease was traced definitely to the presence of certain living organisms occurring in the discharges coming from persons suffering from these disorders. This having once been recognised, the conclusion was not perhaps unnaturally arrived at that a water could not be condemned as dangerous unless it contained some of these pathogenic organisms ; and chemical analysis, being unable to detect these, was condemned as useless. It is because I firmly believe that such condemnation is unwarranted that I venture to bring this subject before you this evening. I need say but little about methods of analysis, but I cannot help drawing attention to the recent remarkable demonstrations of the difficulty connected with the carrying out of the organic carbon and nitrogen process devised by Professors Frankland and Armstrong. Those who have followed the analyses published monthly in the report of the official water examiner must have noticed that while Professor Frankland almost invariably gave the organic carbon higher than did Professors Odling, Crookes, and Tidy, the latter, almost as invariably, gave the organic nitrogen higher than Professor Frankland. The result was that the ratio between organic carbon and organic nitrogen, on which at one time Professor Frankland pinned his faith in judging of the quality of a water was not infrequently twice as high in the one series as in the other. Here, then, we have

on the one hand one of the inventors of the process, a chemist of the highest reputation, whose experience of this method is certainly unrivalled, and on the other two of our foremost chemists, and the most enthusiastic convert to the process, differing month after month, and year after year, in this remarkable manner. What confidence, I ask, can we place after this in the process in the hands of chemists who cannot claim one-hundredth part of the experience of these observers? Quite lately a change has come—in consequence, we are told, of some hints given by Professor Frankland—and the two series of analyses now agree very fairly. If Professors Dewar and Crookes are not above taking a hint from another quarter, I would advise them to discontinue their old unscientific method of estimating the oxygen absorbed from permanganate, and adopt the method now, I believe, very generally employed; namely, the use of stoppered bottles, a temperature of 80° F., and four hours' action. For the purpose of this paper it does not, however, matter what particular method of analysis is adopted, provided it is carried out with care and skill. I will only add on this subject that in my opinion no judgment on any water should ever be formed on one or two analytical features only, but that as many points as possible likely to throw light on the subject should be investigated. For my part, I have for many years been in the habit of giving colour, smell, appearance (clear or turbid), deposit and character of deposit, nitrous acid, phosphoric acid, hardness before and after boiling, total dry residue, colour of the same and behaviour on ignition, oxygen absorbed from permanganate in four hours, sometimes also in fifteen minutes, chlorine, nitric acid, ammonia, albuminoid ammonia, and, only if specially desired, organic carbon and nitrogen. With the help of these data a just conclusion may, I think, be nearly always arrived at.

The next point is, What does such a chemical analysis teach us? It does not, as before stated, show us the presence or absence of pathogenic organisms, or, indeed, of any living organism; and in this respect it is, of course, defective. (Examination of the deposit by the microscope often teaches us a great deal, but is not a chemical examination). But it does show us, in ninety-nine cases out of a hundred, whether the water is polluted by sewage or not, particularly if we adopt the plan proposed by Mr. Hühner and myself—viz., comparing the water under examination with one from the same district and formation which the surroundings show to be unpolluted. No doubt, if a water should be contaminated by a small proportion of, say, a typhoid stool, analysis may not be able to detect this pollution. Such a case, however, very rarely happens, except with artificially-prepared samples; but if it should occur naturally, the analyst would most likely overlook it. In nearly all cases, however, a polluted water will contain the sewage from many healthy persons for that from one person suffering from typhoid, and although the latter by itself could not be detected, the sewage from the larger number of healthy persons can and will be detected. The fact of such pollution having taken place will—and this is a most important point—be detected, however perfect may have been the natural or artificial filtration, and it will of course be detected, whether any pathogenic organisms are present or not.

Now we know that a water polluted by sewage, although at the time of analysis no infected sewage was present, may at any time become dangerous if anyone of the

persons within the drainage area of the well should suffer from typhoid or cholera. The chemist is, therefore, able to anticipate what may happen in the future; and as no one will pretend that a sewage-contaminated water, even if free from pathogenic organisms, is wholesome, the chemist, by advising the closing up of a well or the abandonment of a watercourse, will close, so to speak, the stable-door before the steed is stolen. To put, then, the case of the chemist shortly :

While unable to detect the presence of pathogenic organisms, he is, at any rate in the great majority of cases, able to detect the presence of sewage pollution. He is thereby able to anticipate what may happen in the future, and by timely advice prevent an outbreak of disease.

What, now, can the bacteriologist accomplish? He is, in some cases at least, able to detect the presence of pathogenic organisms in a water, but only *after* the water has become infected. When an outbreak has occurred, he will furnish the final proof that it has been caused by the water—a fact which, quite irrespective of such proof, would in nine cases out of ten have been demonstrated by altogether independent investigation. The bacteriologist, of course, cannot find pathogenic organisms where they are not present, and has not, at least not in the same degree, the power of anticipating the future possessed by the chemist. He is able to close the stable-door, for conclusive reasons, but only *after*, not *before*, the steed has been stolen. I shall of course be told that in a sewage-polluted water the bacteriologist is able to detect numerous organisms non-pathogenic, but, like the bacillus coli, for example, characteristic of sewage. I answer, this may be so; but you condemned chemical analysis on the ground that it cannot detect pathogenic organisms, and you cannot run with the fox and hunt with the hounds. If you acknowledge that sewage-polluted water is not fit for human consumption, whether specially infected or not, you place yourself in the same position as the chemist, and the question will have to be decided, how can sewage pollution be most certainly detected, chemically or biologically? Do not, however, condemn analysis on the ground that it cannot detect pathogenic organisms, and then presume to judge a water, not by the presence or absence of pathogenic organisms, but by the mere number of the micro-organisms present. The proposal to condemn a water because it contains, say, more than a hundred micro-organisms per cubic centimetre appears to me to be simply nonsense. Take a deep well water, Kent water, for example, which in many instances, as it comes from the well, contains as few as ten organisms per cubic centimetre, or even less. Keep this same water for a day or two in moderately warm weather, and it contains thousands in the same volume. Are we to be told that Kent water is an admirable drinking water as it comes from the well, and absolutely unfit for drinking one or two days after?

By means of biological examination we are probably better able to detect pollution in a water contaminated by unfiltered sewage; but this kind of pollution is, in well waters at least, comparatively speaking, a rare occurrence. In most cases the sewage enters the well after filtration through more or less soil, in which many of the organisms remain behind. Experiments with artificially-polluted waters, in which the pollution is brought about by the addition of a small quantity of an infected stool, and without its proper complement of urine, are therefore greatly misleading.

They unduly favour biological examination as compared with chemical analysis. It would be extremely easy to prepare a water highly polluted by sewage, which, on biological examination, would have to be pronounced absolutely free from contamination, but in which chemical analysis would reveal the presence of sewage with the greatest ease. Would biologists consider such an artificially-prepared water a fair sample to judge between biological and chemical examination? I am, of course, aware that there is at least one case on record in which, in all human probability, a single discharge from a person suffering from typhoid, polluting a well, caused a serious outbreak of typhoid. (We shall presently see that, according to Dr. Kelly, the typhoid outbreak at Worthing two years ago was a case of a similar character.) In such a case bacteriological examination might possibly have succeeded in tracing the mischief to the well, while analysis would probably have failed. But biology would only have succeeded when it was too late to prevent mischief. Moreover, such cases are necessarily rare, and never ought to occur; they cannot fairly be brought forward as proving the general superiority of biological examination over chemical analysis. I do not wish to be regarded as hostile to the bacteriological examination of water, but I do not believe that it can replace chemical analysis. In many cases biological examination is, of course, of the highest value. Take the case of our London water-supply. We know that the Thames is largely polluted by sewage, which in many cases is infected sewage, and we do not as yet know sufficient of the life-history of these organisms to be justified in affirming that none will ever reach the intake of the waterworks, and find their way on to the filter-beds. Although, as far as I know, none have ever been detected there, their possible presence must be accepted. The safety of the supply depends, therefore, on the efficiency of the filtration. If, then, it can be shown that filtration is able to remove 99 per cent. of all the micro-organisms present, we are justified in concluding that the chance of pathogenic organisms finding their way into the filtered water is but slight. To judge the efficiency or otherwise of the filtration of such a water, biological examination is certainly a far more effective guide than analysis. But this case also shows the weakness of biological examination. On the strength of it, we are forced to pronounce the water to be pure and free from pollution, whereas the chemist has no difficulty in pronouncing it sewage polluted. Armed with this knowledge, he is able to say: "This water is polluted by sewage, and although not in its present condition injurious, it may at any time, through a breakdown of the filtering arrangements, become dangerous."

Biological examination of water is yet in its infancy; but, judging by the great strides in advance it has made during the last few years, we may confidently look forward to further improvements in the near future. Nevertheless, it will not, in my opinion, ever be able to replace chemical analysis.

Let me draw attention, in passing, to the conveniently small measure adopted by bacteriologists to record their results; namely, the number of colonies yielded by 1 cubic centimetre of water. Now, one organism per cubic centimetre means 4,543 per gallon, or about 300 in an ordinary tumblerful of water. Koch's limit of 100 colonies per cubic centimetre means, therefore, 453,600 per gallon, or 30,000 per tumbler. How beautifully free from impurity would most water, or even sewage,

appear if we stated our analytical results in parts per cubic centimetre, instead of in parts per 100,000, per million, or in grains per gallon. Now, inasmuch as nobody can tell, at present, how small a number of pathogenic organisms may, say under conditions most favourable to their action, produce serious effects, this statement of organisms per cubic centimetre seems very unsatisfactory, seeing that many of us are in the habit of frequently drinking several pints of water a day. No doubt if the bacteriologist finds pathogenic organisms in a water, that water stands condemned, whatever its other qualities may be; but does the converse hold good? Are we justified in pronouncing a water as perfectly safe in which the bacteriologist has been unable to find any such organisms? Can we be sure that the cubic centimetre, or the litre, or the two litres he has examined contained their proper proportion of all the various organisms present in a large bulk of water? Do we know enough of the conditions of existence of these organisms to be justified in such a conclusion? What would be thought of a man who, if he, say, in ten cubic yards of sea-water found no fish, were to pronounce that there are no fish in the sea? or who, when he found a certain number of herrings in ten cubic yards in the midst of a shoal of herrings, would take this as a basis for calculating the number of herrings in the sea? The conclusion, then, that I have arrived at is that each of these two methods of examination has its strong and its weak points, and that neither of them can fully replace the other. They should, if possible, be used in conjunction with each other; but if one kind of examination only can be done, this one should be chemical analysis.

I cannot conclude without making some remarks on the outbreak of enteric fever in Worthing in the spring and summer of 1893, as it offers a good example of the manner in which chemical analysis is often contrasted with bacteriological examinations, to the disadvantage of the former.

Dr. Theodore Thomson says (p. 17 of his report): "Several chemical analyses of 'Worthing' water, before, as well as after, the date on which the new supply was utilized, are given in Appendix B, and from these it will be seen that, as not infrequently happens, chemistry failed to detect any definite impurity in the water. But the result of bacterioscopic examinations, given in Appendix C, furnishes conclusive evidence of contamination of this supply by faecal matter. Let us see how the case really stands. The first case of enteric fever at Worthing was notified on May 3 (as subsequently ascertained, the first attack had occurred on April 16, between which date and May 2 fifteen more persons were attacked). The first sample of water sent to me was drawn on May 2 from just below the surface of the water in the well (well C), while the second sample was taken on May 6 from a depth of 57 feet below the level of the ground, 30 feet below the water-level, and 15½ feet from the bottom of the well. I reported both samples as unpolluted by sewage. At the time I was entirely ignorant of what had happened at Worthing; but even now, with a full knowledge of this, I am still strongly of opinion that these samples, particularly that of May 6, were not polluted by sewage. The latter sample is chemically purer than average Kent waterworks water.

On May 15 another sample of water was taken from the same well (well C), but this time from the bottom, and close to the new heading, through which the pollution was supposed to enter the well. This sample was examined by Professor

Crookshanks, who found it to contain 118,000 bacteria per cubic centimetre. In his report no mention is made either of bacillus coli or the enteric fever bacillus, and as the examination was made with a full knowledge of what had happened at Worthing, we must assume that none were present. On June 5 two more samples were taken from the bottom of well C. One sample was sent to Professor Crookshanks, the other to Professor Millar Thomson. Professor Crookshanks sums up his report as follows: "The water-sample of June 5, 1893, ranks, from the bacteriological analysis, as very pure water." Professor M. Thomson's analysis shows the water to have been, on the whole, not quite as pure as my sample of May 6, but he reports: "On the whole, the chemical analysis of the water sent does not point to the water being unfit for drinking purposes." Here, then, we have three chemical analyses and two bacteriological examinations, none of which point to sewage contamination, the second bacteriological examination being, in fact, highly favourable. Let me repeat here that my second sample—the most important one—was taken 15½ feet from the bottom of the well, and 9½ feet above the crown of the suspected heading. It was, therefore, in all probability, purer than a sample taken from the bottom would have been. Why are these two bacteriological examinations, made by an eminent observer, not mentioned in Dr. Th. Thomson's report? Where, I ask, is the failure of chemical analysis as compared with bacteriological examination, for both methods point to the same conclusion?

On July 26 a further sample of water was taken from well C and sent to Dr. Klein. The depth from which the sample was taken is, unfortunately, not given, but it was probably taken from the bottom of the well. In this sample Dr. Klein detected, apparently, five bacillus coli in the particulate residue from 2,500 cubic centimetre of water, driven through a Berkefeld filter. No enteric fever bacilli were detected in this sample.

On August 2 another sample of water was taken at the Worthing Waterworks, this time drop by drop during twelve hours, from the rising main, through which the water from all the wells, headings, etc., passed. In 1,500 c.c. of this water Dr. Klein detected an abundance of bacillus coli, and three organisms, two on one plate and one on another, "which on sub-culture presented morphologically, as well as culturally, all the characters of the enteric fever bacillus." Observe, Dr. Klein does not say that what he found was actually the enteric fever bacillus, and he was evidently alive to the fact that it might have been some other organism, otherwise why this caution? If a chemist were to state in his report that he found something which gave the reaction of, say, nitric acid, but did not say that it was nitric acid, we should all know what to think of such a statement.

Before finishing this part of the case, it is necessary to say that in Dr. Th. Thomson's report, p. 16, it is clearly shown that under ordinary conditions no sewage would find its way to the near neighbourhood of the heading through which the pollution is supposed to have entered the well, but that when a heavy rainfall coincided with a tide-locked condition of the sewers, some sewage, by being backed up along an old disused overflow pipe, might find its way there. Now, during March and April, 1893, the rainfall at Worthing was 0.33 inches and 0.08 inches respectively, and for seven weeks prior to May 6 practically no rain at all fell. On

the other hand, during June and July the rainfall amounted to 0·58 and 4·95 inches respectively, of which no less than 3·05 inches fell within ten days of June 26 and 0·41 inches on the 26th itself. Where, I ask, is the justification for comparing samples taken on May 2 and 6, at a time of little or no rainfall, and an epidemic only just beginning, with the samples of June 26 and August 2, after heavy rainfall and an epidemic only just past its height, after having run its course for over four months?

If such a conjunction of favourable circumstances is necessary to enable a bacteriologist to detect the presence of three organisms which on subculture presented morphologically and culturally all the characters of the enteric fever bacillus, all I can say is, save us from the bacteriologist!

But the case against bacteriology is not by any means exhausted with regard to this Worthing outbreak.

So far I have only dealt with the outbreak at Worthing and other places, the water-supply of which was taken from the Worthing waterworks. There was, however, a similar outbreak at West Worthing, which has a separate supply. This outbreak was also traced by Dr. Th. Thomson to the water-supply, with this difference: that he believes, from the localization of the outbreak in certain districts of West Worthing, that it was due to local pollution of the water in the mains of these districts, and not to pollution of the water at the waterworks. Here, then, was a fine field for the bacteriologist, who, moreover, as no chemist was employed, had the field all to himself. The result was that Dr. Klein was unable to detect the bacillus coli or the bacillus of enteric fever in either the water taken drop by drop during twelve hours from the rising main at the waterworks, or in the water taken from a tap in direct communication with the water-main in one of the worst stricken streets of West Worthing. It would be highly interesting if we knew what a chemist could have done. What does Dr. Th. Thomson say of this failure of his favourite instrument for water examination? After some remarks I need not quote here, he says: "For specific organisms are usually present in water in small amounts only, and could readily be absent in the small samples selected for examination." Precisely so; this is exactly my view. Any comment of mine is unnecessary. But it is advisable to point out that here we have not only complete failure to detect the pathogenic organisms which must have been present in the supply, but also a total failure to detect the bacillus coli, the organism by help of which mainly the chemist was supposed to have been vanquished. I ask again, where is the justification for extolling bacteriology at the expense of chemical analysis?

In conclusion, it may not be uninteresting to state that, in the opinion of Dr. Kelly, the Medical Officer for Worthing, the outbreak was not due to any sewage pollution of the water in the ordinary sense, but was due, like the case at Caterham, to the misbehaviour of some of the workmen working in the new heading shortly before the outbreak began. Dr. Kelly wrote to me on October 4, 1894: "Chemistry failed to help us because, as a matter of fact, no sewage entered into our new water-supply." My own opinion is, that the greater portion of the epidemic was due to pollution of the water in the mains of the town of Worthing just as in the case of West Worthing.

Dr. Thresh then read the following paper :

**THE INTERPRETATION OF THE RESULTS OBTAINED UPON THE
CHEMICAL AND BACTERIOLOGICAL EXAMINATION OF POTABLE
WATERS.**

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Prior to obtaining a qualification in medicine and in public health, and to becoming a Medical Officer of Health, I had had a considerable experience in the analysis of potable waters, and, like most other analysts, I was accustomed to declare a water good and safe, or impure and dangerous, according to the results of my analyses, basing my opinion upon comparison with standards arbitrarily set up either by analysts of repute, or by learned societies who had given their attention to this subject. Under the conditions of my appointment as Medical Officer of Health, the analysis of drinking-waters formed part of my duties, and as such has led to my having examined probably 1,000 samples of water during the last five or six years.

I was not long in discovering, what other Medical Officers of Health had previously discovered, that my analytical results often directly contradicted the results of my observations as to the source of the water, and its effects upon the health of those who used it. My standards condemned waters as impure and unsafe which had never been known to produce any ill effect, and which came from sources which, so far as I could discover, were perfectly safe and free from any injurious contamination. In fact, if I were to condemn every source of supply in my districts which yielded an excess, for example, of albuminoid ammonia and nitrates, I should leave many localities entirely without a supply. On the other hand, waters which I have had the gravest reasons for suspecting as the cause of disease, yielded on analysis much more favourable results than others not suspected, and yet upon close observation such waters were found either actually contaminated with sewage or liable to intermittent pollution.

Further experience has also unfortunately brought me in direct conflict with analysts who have differed from me in opinion as to the quality of a water. The analyst judging from the results of his examination only; I basing my opinion not only on the analysis, but also upon an examination of the source of supply, to ascertain the possibility or otherwise of its contamination. My experience, I am sorry to say, is that of other health officers, and it is my desire to avoid such conflict of opinions in future that has induced me to read this paper. Let it be understood that I am not now referring to difference of opinion arising from crass ignorance, as in a case recently submitted to me, in which a deep well water containing a considerable amount of chlorides was stated to be probably polluted with urine. Had the analyst known anything of the nature of the water derived from the source from which this came, he would not have made such an absurd statement.

Of the many impurities which drinking-water may contain, the organic matter only is a serious source of danger, and by far the greatest risk is incurred in using waters liable to contain certain living organisms which, when introduced into the

system, are capable of producing specific disease. Of the presence or absence of such organisms chemical analysis can give us no information. The presence of organic matter may be chemically demonstrated, but inasmuch as the nature of this organic matter, whether poisonous or innocuous, is beyond the power of the analyst to reveal, it is obvious that the results of a mere chemical analysis may often be worthless or even misleading. This point cannot be too strongly emphasized, since the popular impression, shared alike by the ignorant and the learned, that a chemist, by performing a few mysterious experiments with a water in his laboratory, can pronounce at once whether it be safe or dangerous, must be dispelled. This opinion has been fostered by those analysts who do not hesitate to pass judgment upon a water merely from the results of their chemical examination, from the determination of the chlorides, nitrates, phosphates, and ammonia, of the organic carbon and nitrogen, and of the oxygen consumed, or of the ammonia derivable from the organic matter. All these factors are of more or less importance as an index of the degree of pollution, recent or remote, but their real value can in very few cases be assessed without some previous knowledge of the source of the water. The inorganic constituents can easily be determined, and whether, either in quantity or quality, these are objectionable, the chemist can safely express an opinion. But this is more medical than chemical. For our purpose, therefore, only those salts need be considered which by their presence tend to throw some light upon the source of the organic matter, always present in greater or less quantity. These are the chlorides, phosphates, nitrites, nitrates, and ammonia, and inasmuch as their determination is often of importance, the value of each may be discussed.

Chlorides.—In the great majority of instances the only chloride present is chloride of sodium or common salt; occasionally other chlorides (calcium, magnesium, etc.) are also found, especially if the water be derived from wells near the sea-coast. Rain-water always contains a trace of salt, the amount varying with the distance from the sea. Certain geological formations also are rich in salt, and waters obtained therefrom may contain considerable quantities of it. Urine, again, contains nearly 1 per cent. of salt; hence pollution with sewage will add chlorine to the water. The effluents from many manufactories, alkali works, mines, etc., are also rich in chlorine. From these various sources, therefore, the chlorides found in waters are derived. When the geological strata contain little or no salt, and there are no manufacturing or mining effluents to pollute the water, the amount of chlorides present may serve roughly as an index of the extent to which it has been contaminated by sewage.

In Massachusetts it has been found that the amount of chlorine in the surface waters and streams decreases in amount from the seaboard westward or inland. By the examination of waters from sources removed from all risk of contamination, the normal chlorine for such districts has been determined. "By placing on the map of the State the amount of chlorine normally present in its unpolluted waters, and then connecting the points of equal amounts, lines of like chlorine contents are obtained, which are called *isochlors*." From the map thus prepared the normal chlorine is found to vary from .45 grain per gallon near the coast to less than .06 in the western part of the State (Board of Health Report, 1892). Over any given area the amount of chlorine in excess of the normal, as above ascertained, can only

be due to the influence of the population discharging its sewage thereupon. Assuming that 100 persons per square mile add on an average .03 grain per gallon of chlorine to the water flowing from that area, the extent of the contamination can be approximately calculated. It must be remembered, however, that the amount of chlorine present does not necessarily signify present pollution. The organic matter which originally accompanied the salt, and which alone is deleterious, may have undergone complete oxidation and destruction, so that organically the water may be very pure, although the amount of chlorine present indicates that at one time it was excessively polluted. This fact detracts very considerably from the importance of the chlorine determination. It affords some evidence of the previous history of the water, and that is all. In insular countries the estimation of the chlorine is of even less value, since they cannot be mapped out into *isochlors*. Over limited areas, however, the normal chlorine may sometimes be ascertained, and any excess found in samples from that district will be in a measure proportionate to the present or past pollution of the water. For example, in the parish of Writtle, all the wells in which I recently examined, the normal chlorine did not exceed 2.5 grains per gallon, yet in that parish subsoil waters were found containing as much as 14.0 grains per gallon, and that this was due to past and present pollution with sewage was substantiated by the excess of other substances, especially nitrates, which, as we shall see, are also in most cases derived from the same source. Unless this normal chlorine be known, the determination of the chlorides has no value whatever. The variation in the amount of chlorine in pure surface waters from various geological formations being known, any excess over these amounts would probably point to past or present pollution, and in any case would indicate that farther investigation of the source was desirable or necessary. In shallow-well waters, even when pure, the chlorine varies so greatly in amount that it is only in rare cases, as in the one referred to above, that the determination affords any information of value. In spring waters also it is difficult to decide upon the normal chlorine of any particular formation, but if in any case the amount found greatly exceeds the average, past or present pollution is indicated. The same remark applies to deep-well waters. In several instances, when examining water from the deep wells in Essex, I have found that the chlorides present were below the normal, and have been able to prove that this was due to surface water (usually impure) having gained access to the well. If the source of the water be not known, reliance upon the chlorine estimation may lead to serious error. The possibility of the excess of chlorine being derived from manufactories or mines must also be considered before concluding that the water contains contaminating matter of animal origin.

Nitrates and Nitrites.—The combined nitrogen found in drinking-waters may be present as organic matter or in ammonia, nitrites, and nitrates. Traces of all three are found in most samples of rain-water. Nitrogenous organic matter undergoing putrefaction invariably produces ammonia, and by oxidation this ammonia is converted, by micro-organisms found in all soils, into water and nitric acid, the latter decomposing the carbonates present, and forming nitrates of soda, potash, or lime. The ammonia, however, is not apparently converted directly into nitric acid, but passes through an intermediate stage, a lower oxide of nitrogen, nitrous acid being

first formed. The Rivers Pollution Commissioners found that whilst the organic matters contained in sewage, and therefore of animal origin, yielded abundance of nitrates and nitrites by oxidation, no less than 97 per cent. of the combined nitrogen of London sewage being converted into nitrates by slow percolation through 5 feet of gravelly soil, vegetable matters yielded mere traces of these compounds. Upland surface waters "in contact only with mineral matters, or with the vegetable matter of uncultivated soil, contain, if any, mere traces of nitrogen in the form of nitrates and nitrites; but . . . as soon as the water comes in contact with cultivated land, or is polluted by the drainage from farmyards or human habitations, nitrates in abundance make their appearance." Subsoil waters derive their nitrates in part from the oxidized ammonia of rain water, in part from the slow decay of vegetable matter, and in part from sewage matters. The amount derived from the two former is almost invariably small. Vegetable matter is not highly nitrogenous, and as a rule decomposes but slowly. Animal matter, on the contrary, decomposes rapidly and yields much ammonia. Nitrates serve for the food of plants, and the active growth of vegetation may remove nearly the whole of these salts from a water. In reservoirs the nitrates decrease gradually as the vegetable organisms increase. The total combined nitrogen, therefore, in a water may at one time exist in decaying animal and vegetable matter, or in the form of ammonia; at another in the form of nitrites and nitrates, and yet again as a constituent of the protoplasm of living vegetable organisms, in which latter case it is not in solution, but merely suspended in the water. Whenever organic matter undergoes putrefaction in the absence of air or free oxygen, not only are nitrates not formed, but any nitrates present are decomposed, their oxygen being required for the formation of water and carbonic acid by combination with the carbon and hydrogen of the decomposing substances. The nitrogen appears to be set free, possibly accounting for the excessive amount of that element found in such deep-spring waters as those of Bath, Buxton, and Wildbad. In this way the small amount of nitrates found in most deep-well waters is accounted for. Such being the case, it is evident that even concentrated sewage may undergo such changes as would totally obscure its origin so far as the combined nitrogen is concerned. At first it would be contained chiefly in the dissolved animal impurities; after passing through the surface soil, it would exist chiefly in the nitrates formed by the oxidation of the organic matter; later the nitrates may be decomposed, and the nitrogen liberated, when the water would be almost or entirely free from combined nitrogen. On the other hand, certain deep-well waters, especially in the chalk, contain very considerable amounts of nitrates, which it is difficult to believe are derived from the oxidation of sewage matters. It has been suggested that these nitrates are derived from fossil remains, or from natural deposits of nitrates, or from vegetable matter; but as no proof of these statements is forthcoming, they must be received with reserve. In the eastern counties the chalk wells yield waters which in some districts are absolutely free from nitrates (south-east Essex), whilst in other districts (Norfolk) they may contain possibly as much as 1 grain of nitric nitrogen per gallon. The following may be quoted as examples:

	Nitric N. per gallon.	Depth of Well. feet.	Authorities.
Stratford: Phoenix Works	·00	200	J. C. Thresh.
Wimbledon... ..	·03	200	"
Chatham Public Supply	·48	490	"
Southend "	·05	900	"
Witham "	·45	600	R.P.C.
Mistley: Tendring Hundred W. W. Co. .	·05	160	J. C. Thresh.
Braintree Public Supply	·02	430	T. A. Pooley.
Colchester (Donyland Brewery)	·00	305	J. C. Thresh.
Saffron Walden Public Supply	·95	46	"
Norwich	·80	About 400	"

In none of the above examples is there any possibility of recent sewage contamination.

Notwithstanding these facts, the Rivers Pollution Commissioners considered the total combined nitrogen to be an index of previous sewage contamination. They assumed that 100,000 parts of average London sewage contains 10 parts of combined nitrogen in solution. The mean amount of such nitrogen found in a large number of samples of rain-waters examined was ·032 per 100,000. After deducting this latter amount from the amount of nitrogen, in the form of nitrates, nitrites and ammonia found in 100,000 parts of a potable water, the remainder, if any, they say, "represents the nitrogen derived from oxidized animal matters, with which the water has been in contact. Thus, a sample of water which contains, in the forms of nitrates, nitrites, and ammonia, ·326 parts of nitrogen in 100,000 parts, has obtained $\cdot326 - \cdot032 = \cdot294$ part of that nitrogen from animal matters. Now, this last amount of combined nitrogen is assumed to be contained in 2,940 parts of average London sewage, and hence such a sample of water is said to exhibit 2,940 parts of previous sewage or animal contamination in 100,000 parts." The Rivers Pollution Commissioners, however, point out that, on the one hand, the nitrates may not indicate the full extent of the previous sewage pollution, since the roots of growing crops take up much of the ammonia, nitrites, and nitrates contained in polluted water, and animal matter, which decomposes without access of air, destroys nitrates; and, on the other hand, that the nitrates present may indicate 10 per cent. of previous sewage contamination in deep wells and springs, and the risk of using such waters be regarded as nil, providing surface pollution be rigidly excluded. This 10 per cent. of previous sewage contamination corresponds to one grain of nitric nitrogen per gallon.

Mr. F. Wallis Stoddart, in an excellent paper on 'The Interpretation of the Results of Water Analysis,'* describes a series of experiments in which he passed sewage containing cholera bacilli through a nitrifying bed of coarsely-powdered chalk, and found that although the organic matter in solution was completely nitrified, yet the cholera bacilli or spirilla could be detected in the effluent. From the result of his own observations and experiments, he concludes that natural waters "can at most obtain from one-tenth to two-tenths of a grain of nitrogen as nitrates per gallon from

* *Practitioner*, 1898; reprinted in *ANALYST*, xviii., 293; xix., 18, 89.

sources other than animal matter," and "that practically the whole of the nitrogen of sewage may be oxidized into nitric acid without materially diminishing the risk involved in drinking it." He urges that whenever the nitrogen as nitrates exceeds half a grain per gallon, it indicates "either dangerous proximity of the well to a source of pollution, or such easy communication with it that the distance separating the two points is no guarantee of purification." Many waters, the source of which I carefully examined, and which were collected and analysed by myself, contain more than this amount of nitric nitrogen; and I am perfectly convinced that these waters are hygienically of the highest class, and can be used without the slightest risk or danger. On the other hand, in the table given at the end of this paper, there will be found many waters containing very much less nitrogen as nitrates which have almost certainly (in most cases the proof was most conclusive) given rise to outbreaks of typhoid fever. If Mr. Stoddart's maximum of $\cdot 5$ be accepted as proof that a water is dangerous, then the public and private water-supplies of many of our healthiest districts—districts remarkably free from outbreaks of typhoid fever—must all be considered dangerous. As a matter of fact, the amount of nitrates which would condemn a water from one source may be absolutely without significance in another, all of which goes to prove, as will be shown in the sequel, that mere chemical analysis is absolutely powerless to prove that any water is of such a quality as to be incapable of producing disease amongst those who drink it.

Nitrites may result from the oxidation of ammonia, or from the reduction of nitrates, and, as it is an easily oxidizable compound, its presence indicates a condition of instability, of matter undergoing change. Usually this matter is of animal origin and derived from manure or sewage, the ammonia produced by their decomposition being in process of oxidation to nitrates. Where the soil is not sufficient in quantity, or is defective in quality, the oxidation may be incomplete, and incompletely purified and probably incompletely filtered water is the result. Usually, in such cases an excessive amount of ammonia is also present. But, though usually, this is not invariably the source of the nitrates and ammonia. Where nitrates are present the nitric acid may be reduced by contact with metals, such as iron or lead, forming the pipes in which the water is conveyed, or lining the upper portion of the well. Where such is the case, a trace of the metal can always be detected in the water. Unless this fact be borne in mind—and it often appears to be overlooked—a good and wholesome water may be classed as dangerous or polluted. Certain organisms also found in water are capable of reducing nitrates to nitrites. Still, the presence of nitrites always renders a water suspicious, and their origin should be carefully investigated.

Ammonia.—All rain-water contains this compound, as does also melted snow. The first portions of a shower, and the rain collected in the neighbourhood of towns are richest in ammonia. As an average, $\cdot 02$ grain per gallon, taken by the Rivers Pollution Commissioners, is probably fairly approximate, but the variation is very wide ($\cdot 2$ to $\cdot 01$). In passing over or through the ground the ammonia is rapidly oxidized, and by the time the water reaches a stream or the general body of subsoil water, most of it has disappeared. Rain-water stored in covered cisterns, however, usually retains its ammonia for a considerable period. In such waters, therefore, the ammonia, unless excessive, is devoid of significance. Many deep-well waters also

contain much ammonia, the origin of which has given rise to a good deal of surmise. The generally-accepted theory is that it is due to the reducing action of ferruginous sands on the nitrates present. This may be so in some cases, but my observations lead me to believe that it is often due to the reduction of the nitrates by the metal of the bore tube, pump pipe, and lining of the well. I was led to this conclusion from the fact that I found the water from one and the same well at one time quite free from ammonia, and at another containing as much as one part of ammonia per million parts of water. In the water containing ammonia I also found a very faint turbidity, which cleared up on the addition of a little acid, and gave the reactions for iron. The clear, ammonia-free water also, when stored for a time in an iron tube, became turbid, and nitrites, ammonia and iron could be detected in it. Generally, however, the ammonia found in river, spring, and well waters is derived from putrescent animal matter—that is, from manure and sewage; but before this conclusion can be safely drawn, the other possible sources must be excluded. Dr. Brown, in his *Report to the Massachusetts State Board of Health*, 1892, whilst agreeing that imperfect oxidation of sewage matter is generally the source of the ammonia, calls attention to the fact that several waters in the State free from such pollution contain a considerable amount of free ammonia. "They are all associated with iron oxide and the fungus *Crenothrix*." Such waters are found also in many swampy regions, and in wells sunk in ferruginous river silt, and usually become turbid from the formation and deposition of oxide of iron when exposed to the air. The odour of these waters is said to be "often disagreeable from dissolved sulphuretted and carburetted hydrogen."

Phosphates.—Phosphates are found widely distributed in rocks and soils, and traces may be dissolved by a water rich in carbonic acid. Albuminous matters, whether of animal or vegetable origin, give rise to phosphates by their decay, hence an excessive amount present in a drinking-water is held by many analysts to indicate contamination. The difficulty of determining their presence and amount is considerable, and as the value of the determination is very doubtful, few analysts pay any attention to them. They may be present in a pure water, and their absence is no proof that a water is free from pollution.

Organic Matter.—By no known process can either the quantity or quality of the organic matter in water be determined. When a known volume of water is evaporated to dryness, the weight of the residue is that of the inorganic and organic substances contained therein. When this residue is ignited the organic matter is destroyed and volatilized, and the "loss on ignition" has been regarded as approximately expressing the weight of the organic constituents. Such, however, is rarely the case, since carbonic acid may be driven off from the carbonates present, and any nitrates present will be more or less completely reduced. Moreover, some salts retain water so tenaciously that the whole is not driven off at the temperature used for drying, and this moisture is given off when the residue is ignited. For these reasons, chiefly, the "loss on ignition" cannot be depended upon as an index of the amount of organic matter present. But although the total amount of the animal and vegetable substances cannot be determined, the carbon and nitrogen therein can be ascertained by careful chemical analysis. Not only so, but the authors of the original process believed that, with certain reservations, the propor-

tion of the nitrogen to carbon indicated whether the organic materials are derived from the animal or vegetable kingdom. In fresh peaty water the Rivers Pollution Commissioners found that $N : C = 1 : 11.93$, whilst in similar waters, which had been stored for weeks or months in lakes, $N : C = 1 : 5.92$. After such water had been filtered through porous strata, $N : C = 1 : 3.26$. In fresh sewage the average of a large number of samples gave $N : C = 1 : 2.1$. Highly polluted well-waters, soakages from cesspools, etc., gave $N : C = 1 : 3.126$. In sewage after filtration through soil the proportion of N to C rose from $1 : 1.8$ to from $1 : 4.9$ to $1 : 7.7$. Evidently, therefore, the ratios of N to C "in soluble, vegetable, and animal organic matters vary in opposite directions during oxidation—a fact which renders more difficult the decision as to whether the organic matter present in any given sample of water is of animal or vegetable origin."

All nitrogenous organic matter, whether of vegetable or animal origin, yields more or less ammonia when boiled with a strongly alkaline solution of permanganate of potash, and the ammonia so yielded by potable waters is called "albuminoid," or "organic" ammonia. The proportion of nitrogen in the ammonia so yielded to the total nitrogen in the organic matter is unfortunately not constant; but the chemists to the Massachusetts Board of Health believe that when the process is performed as in their practice, about one-half of the nitrogen is converted into ammonia. Albuminoid substances of animal origin contain about 16 per cent. of nitrogen, but vegetable matters contain very much less; hence the amount of "albuminoid" ammonia is no index to the amount of organic matter present in the water. Mr. Wanklyn, who devised this process, considers that undeniably contaminated waters always yield an excessive amount of albuminoid ammonia (over .10 part per million); usually with much free ammonia (over .08 part per million).

If the albuminoid ammonia distils over very slowly and is in excess, but the water contains little free ammonia and very small quantities of chlorides, Mr. Wanklyn considers this an indication that the contaminating matter is of vegetable origin. He adds: "The analytical characters, as brought out by the ammonia process, are very distinctive of good and bad waters, and are quite unmistakable." The generally accepted opinion, however, is that no reliance can be placed upon these determinations taken alone, and in the *Massachusetts State Board of Health Report* for 1890 there is quoted, as an example, the results of the analyses of certain of the Boston water-supplies. Reservoir No. 4 is known to contain the purest water, but the average "albuminoid ammonia" during two years was .26 per million. The water of the Mystic Lake is the worst of the Boston waters, since it contains both sewage and manufacturing refuse; yet during the same period the average albuminoid ammonia was exactly the same as in the purer water. In the table appended many other examples will be found of the erroneous conclusions which may be drawn from a too implicit reliance upon the determination of the ammonia yielded by distillation with alkaline permanganate.

Forschhammer devised a process for the estimation of the amount of oxygen required to oxidize the organic matter in water. This method, as improved by the late Dr. Tidy, has become very popular, and many attempts have been made to render the results comparable with those obtained by Frankland's process, in which

the amount of organic carbon and nitrogen is ascertained by combustion, but with only partial success. The results, when compared with those obtained by the "albuminoid ammonia" process, prove that there is no relation between the amount of ammonia yielded by a water when distilled with an alkaline solution of permanganate of potash, and the amount of oxygen absorbed when the water is digested with an acid solution of the same salt. This process tells us little or nothing of the nature of the polluting material; it does not even distinguish between organic matter of vegetable and animal origin, and it affords us no evidence of the amount of such substances present. The presence of certain bodies of mineral origin (sulphuretted hydrogen, nitrites, the lower oxides of iron, etc.) also cause absorption of oxygen, and unless great care is taken to ascertain the absence of these, or to ascertain the exact amount of oxygen consumed by them if present, serious errors may be introduced. When these corrections are made the oxygen process is still open to all the objections which have been urged against the albuminoid ammonia process. It may condemn a perfectly harmless water as polluted, and pass as of good quality a water of most dangerous character. The following table was devised by Drs. Tidy and Frankland:

AMOUNT OF OXYGEN ABSORBED BY 1,000,000 PARTS OF WATER.

		Upland Surface Water.	Water other than Upland Surface Water.
Water of great organic purity	...	Not more than 1·0	Not more than ·5
„ medium purity	...	„ „ 3·0	„ „ ·15
„ doubtful purity	...	„ „ 4·0	„ „ ·2
Impure water	...	More than ... 4·0	More than ... 2·0

When the quality of a water is considered from the biological side instead of the chemical, the absurdity of dividing waters into classes of pure, medium, doubtful purity, and impure, is obvious. A water containing a poisonous amount of typhoid bacilli might upon analysis be brought within any of these classes, according to the quantity and quality of the accompanying impurities. In the analyses given below there are instances of waters coming within Tidy's limit of "great organic purity," yet which proved to be capable of causing disease. I have examined many such waters myself, and have also passed many waters as perfectly safe for domestic purposes, which a mere reference to the above standards would have condemned as doubtful or impure.

Many other special processes for determining whether a water be safe or dangerous have been devised, but inasmuch as they are rarely used, it may safely be inferred that they possess no advantage over those to which we have already referred.

Sir Charles Cameron, in his "Address on Public Health," delivered before the British Medical Association at Bristol (1894), says: "We have not yet determined, in relation to chemical analysis, the line which divides a bad from a good water, nor

do I believe that such a line can be drawn. One that contains very little organic matter may be dangerous to drink, whilst, on the contrary, water with a large amount of organic matter has been drunk with impunity for years. It is unfortunate that there is no accurate chemical method for determining the precise nature of the organic matter found. As a rule, the larger the percentage of nitrogen contained in the organic matter, the more likely is it to have been derived from an animal source. Analysis certainly shows the amount of organic matter present, and although such matter is usually innocuous, it is sometimes the reverse. The greater the amount of organic matter, or rather of albuminoid matter, the greater the chance that it may include infective substance. When we have a water with a very small amount of nitrogenous organic matter, there is no likelihood that typhoid dejections will be present. Under the ordinary conditions affecting our water-supplies, it is improbable that typhoid stools would be mixed with them. They are thrown into water-closets, privies, ashpits, manure heaps, the soil, ditches, rivers, etc. From all these media they may find their way into rivers or wells furnishing potable water, but with rare exceptions there must, under such circumstances, be large quantities of other kinds of organic matter associated with the typhoid excreta, and carried with it into the water. In short, the passage of unmixed excreta into potable water must be a circumstance of rare occurrence. In the ordinary cases of pollution by typhoid or other dejecta, the analysis of water proves its unfitness for use."

The latter statement is directly contradicted by the results of the chemical examination of very many samples of water which have given rise to epidemics of typhoid fever. A reference to the table already referred to will show that many most extensive epidemics have been attributed to the use of waters which chemists of eminence had declared to be of a high degree of purity. In these cases also repeated analyses failed to prove "unfitness for use."

Whilst no single determination will enable the analyst to certify that a water is free from danger, or that it is so polluted as to be dangerous to health, the determination of several constituents may enable him to pronounce it to be polluted and dangerous, but will never justify him in certifying that it can be used absolutely without risk. As the freedom from all dangerous polluting material is the information usually sought from the analyst, it follows that if this cannot be ascertained by analysis, a chemical examination is in most cases quite useless. Where a water is known to be contaminated with sewage, or known to be liable to such pollution, an analysis is superfluous. When we also consider that many sources of supply are only subject to intermittent pollution, and that waters from the same reservoir or from the same well (*vide* Analyses Nos. 24, 25, and 26, 27) may vary considerably in composition, according to the depth from which the samples are taken, the character of the season, etc., it is obvious that the chemical examination of a water is a matter of comparatively trifling importance compared with the thorough examination of the source of the water and a knowledge of its history. Frequently waters are sent for analysis, and the analyst is wilfully kept in ignorance of their source, lest the information should prejudice his report, yet without this knowledge he is not justified in expressing the opinion that the water from any source can be used with safety. In commenting upon a recent paper in which I expressed these views, a writer in

the *Chemist and Druggist* says : " It would seem, therefore, that we are face to face with the question, 'Is water analysis a failure?' It has been so exclusively the province of chemical analysts to pronounce judgment upon domestic waters, and they generally have given so little attention to the large issues attached to analysis, and so very much to sets of standard figures for chlorine, nitrogen, hardness, and so on, that the attack from the medical health side is not unexpected. There has been more wrangling over water analyses than over anything else in chemistry—and for what? Some figure in the second or third place of decimals, probably, and in regard to what this ammonia or that ammonia implies, when a visit to the source of the water and an inspection of the sewage trickling into it might settle everything. That is what Sir George Buchanan and Dr. Thresh advocate." The Royal Commission on Metropolitan Water Supply received evidence proving that waters containing very large amounts of organic matter were drunk continuously by a population with perfect impunity, whilst other waters containing so little organic matter as almost to defy chemical detection had proved, time after time, to be of the most poisonous character. For these reasons they conclude that the water question has passed from the domain of chemistry into that of biology. This, however, is not strictly correct. The biological problems involved in the investigation of water supplies are numerous and complex, and as yet but imperfectly understood. At the present time it is doubtful whether a biological examination really tells us more than a chemical analysis, and very often it cannot tell us as much. The reason will be explained shortly.

With the discovery of the fact that such diseases as typhoid fever and cholera are due to the introduction into the system, not of dead organic matter, but of actual living organisms, faith in the chemical analysis of waters began to be shaken. When still more recently, the actual microbes causing these diseases had been identified, and processes had been devised for isolating them from the multitude of other organisms found in water, it seemed as though the examination of water for sanitary purposes had passed from the domain of the chemist to that of the bacteriologist. The study of the number and character of the bacteria, it was hoped, would enable the biologist to definitely pronounce whether a certain water was capable of causing disease, or whether it was perfectly harmless in character. Up to the present time such hopes have not been realised, and the results of an ordinary bacteriological examination are as likely to be misleading as those of a chemical analysis. The reason for this is not difficult to explain, when the significance of certain of the discoveries made by bacteriologists is thoroughly understood. An enormous number of species of bacteria have already been discovered, although the science is in its infancy. They are almost ubiquitous, abounding in the air, water, and nearly all articles of food and drink. Of this immense variety very few appear to be capable of causing disease; the remainder are perfectly harmless to human beings, whilst many are already known to discharge most important functions in the economy of nature. Upon their presence the fertility of soil in a great measure depends; they break down the dead organic matter into the simpler forms which can be assimilated by the roots of plants. By their action the foul organic constituents of polluted water are converted into carbonic and nitric acids, which, in combination with the mineral

bases, form innocuous carbonates and nitrates. They are, in fact, nature's scavengers, consuming the foul and effete, and producing therefrom food for the living.

[The remainder of this paper, and the discussion which ensued on it and the preceding paper, will be published in our next issue.]

Purification of Streams by Green Plants, by Bacteria, and otherwise.—

Researches on this question by Dr. T. Bokorny (*Arch. f. Hygien*, 1894, xx., No. 2) and Duclaux (*Ann. Inst. Pasteur*, 1894, viii., pp. 117-127) are abstracted in *Royal Micro. Soc. Journ.*, 1894, p. 714. Bokorny remarks on the well-known fact that green plants can assimilate organic substances, and the apparent certainty that aquatic bacteria play a very important part in the purification of water containing a considerable quantity of organic matter; and then recounts his own experiments, chiefly with algæ, and especially with diatoms. Volatile fatty acids, amido-acids, indol, skatol, phenylacetic acid, urea, etc., were exposed in suitable solutions—(a) with free access of light, and (b) with exclusion of light—to the action of algæ. The results showed that algæ were capable of using as nutriment volatile fatty acids, such as butyric and valerianic, and also glucose, leucin, and tyrosin. Even urea, when properly diluted, could be assimilated. It seems clear, therefore, that many organic impurities that are drained into rivers are destroyed by water-plants as well as by bacteria.

Duclaux's work concerns the purification of river-water by bacteria, and its "spontaneous purification," only. After alluding to the condition of the Seine above Paris, and the quantity of impurities deposited in the river from the city, he shows that lower down the river has again become practically pure. He quotes Prausnitz's researches on the water of the Isar, which, when entering Munich, contained 305 germs per c.c., but seven kilometres below the city 12,600 per c.c.; whilst thirty-three kilometres below Munich (eight hours' flow) the number of germs had been reduced to one-sixth. The causes of this purification, according to Duclaux, are both physical and vital—particularly notable among the former being the action of the ground-waters, which mingle with the stream along its course, and which influence purification, partly from their lower temperature in summer, partly from their chemical composition. Another prominent physical mode of purification is the deposition of floating particles, all of which, on account of their density, tend to fall to the bottom and drag down other matters with them, this action being aided by the molecular adhesion of water and the particles; but the purification is effected more surely when an organic or mineral precipitate is formed than when these bodies are left simply to the action of gravity or of molecular adhesion. The principal agent in the purification is, however, the microbe; the rôle of the physical agencies being subsidiary thereto.

F. H. P. C.

On the Micro-Chemical Examination of Quinine. H. Behrens. (*Recueil des Travaux Chim. des Pays-Bas*, xiii., 1, pp. 1-12.)—For the detection of impurities in quinine a series of crystallizations is employed, and the form of the crystals examined under the microscope:

1. *Crystallization of Quinine Bisulphate*.—From 0.05 to 0.2 grammes of the impure sample is warmed with two parts of water, and sufficient H_2SO_4 to retain three-quarters in solution, and crystallized at as low a temperature as possible. If less than two-thirds of the solid taken be obtained, the mother liquor must be slightly concentrated on the water-bath and cooled. The crystals obtained are pure bisulphate, while the other alkaloids are left in the mother liquor. The crystals are separated by pressure with a platinum spatula, and washed with several drops of water.

2. *Crystallization of Normal Quinine Sulphate*.—Small quantities of NaHCO_3 solution are added to the mother liquor with warming, and shaking until neutral, leaving the liquid clear. On cooling a large quantity of the sulphate is deposited in long needle-shaped crystals, which are separated from the mother liquor as before.

3. *Second Crystallization of Normal Sulphate*.—The neutral mother liquor is evaporated to dryness over H_2SO_4 , or over a water-bath, larger crystals being obtained by the former process. The crystals dissolve very slowly in cold water, while the sulphates of quinidine, cinchonine, and cinchonidine may be extracted by adding several drops. A small quantity of this liquid may be warmed, a drop of NaHCO_3 solution added, and the crystals formed examined. Lumpy crystals or rhombs point to the probable presence of quinidine, short needles to cinchonine and cinchonidine, while a powdery deposit, changing on boiling into microscopic drops, denotes a considerable residue of quinine. In the last case the quinine should be removed by precipitation with potassium oxalate before proceeding.

4. *Fractional Precipitation*.—Quinidine is first removed by adding a saturated solution of KI, which forms a cloudy precipitate in a few seconds. Cinchonine and cinchonidine are not precipitated by this reagent. After removal of the greater part of the quinidine, fractional precipitation is employed. The liquid, warmed to 40°C ., is stirred with a platinum wire dipped in NaHCO_3 solution, and the deposit examined under the microscope. If rectangular rod crystals of cinchonine are observed on cooling, they should be left a few minutes to develop. If outside these there are needle-shaped crystals of cinchonidine, the whole should be warmed several times to boiling, the evaporated water being replaced. After this, large well-developed crystals of cinchonine may often be found at the edge of the drops. When undoubted crystals of cinchonine have been obtained, the mother liquor is allowed to cool at one side, and the cinchonine crystals fixed to the glass washed with a drop of water. A second precipitation is then made in the same way, when mixed crystals are often obtained, some of them being the characteristic needles of cinchonidine. Only in the presence of a large quantity of cinchonine is a third crystallization necessary.

The author employed the method successfully in the examination of a mixture consisting of 0.1 gramme impure quinine sulphate, containing 0.5 per cent. cinchonine, 0.3 per cent. cinchonidine, and 0.2 per cent. quinidine.

C. A. M.

The Analysis of Varnishes. Parker C. McIlhiney. (*Jour. Amer. Chem. Soc.*, 1894, 848.) After criticising the various methods which have been hitherto proposed for the separation and estimation of the turpentine and other volatile constituents of a varnish, the author proposes the following. It is based on the well-known fact that on distilling a mixture of two immiscible liquids the boiling-point is lower than that of the more volatile constituent. On distilling together 100 c.c. of water and 5 c.c. of turpentine, the first 95 c.c. of the distillate contains all the turpentine, which readily separates from the water. Experiment showed that 90 c.c. of water either dissolves or holds in suspension about 0.3 gramme of turpentine. On these principles the following process was devised: Twenty-five grammes of varnish are weighed into a flask of 400 c.c. capacity, in which has been placed a piece of granulated tin or its equivalent to prevent bumping, and 10 c.c. water. The contents of the flask are now submitted to distillation, the distillate being caught in a tapped separator. When from 90 to 95 c.c. of water have come over, the distillation is stopped and the turpentine allowed to separate. Should the contents of the flask still retain the odour of turpentine, more water is added and the distillation resumed. The separated turpentine is weighed in a tared flask, and a correction of 0.3 gramme for every 90 c.c. of water made. The results obtained were satisfactory. In some experiments salt was added to the water, but no apparent advantage was gained, except when the non-volatile residue was heavier than water. In this case the salt prevented the residue from sinking to the bottom of the flask, and causing bumping by greasing the flask. If it is desired to examine the residue, the remaining water is poured off, alcohol added, this distilled off, and the last traces of alcohol removed by evaporation with a little ether. The residue is not heated above the boiling-point of water; the turpentine is weighed as such, and may be subjected to further examination. For the determination of petroleum products in turpentine, the method of Burton gives the best results. It depends upon the conversion of the turpentine into acids soluble in water by the action of nitric acid, while the other products remain unaffected.

The analysis of the residue, which consists of linseed oil and one or more resins, all more or less altered by heat, presents an extremely difficult problem, which as yet remains unsolved. The author has applied to these residues the same tests as have been applied to oils—viz., the acid figure, the Kœttstorfer figure, the iodine and bromine figures, the bromine addition figure, and the solubility in ether, alcohol, etc.* He arrives at the following conclusions: The differences in the solubility between linseed oil and melted resins are but slight and of little value in effecting separations. The Hübl figure is evidently useless as a means of quantitative analysis, for the figures of linseed oil and kauri, the most frequently occurring resin, are almost identical. The difference in the Kœttstorfer figure is only about 75, and even supposing that the figures of both oil and resin are accurately known, which is not often the case, it would be difficult to make the analysis so carefully that the percentages of oil and resin would be correct. The acid figure also fails when applied to a varnish, though it proves correct with a mixture of common resin and linseed oil which has not been heated too long. The bromine absorbed is of no more

* A long list of these figures for a large number of resins, and the corresponding ones for raw and boiled linseed oil, are given in the paper.

use than the Hübl figure, for its quantity depends largely on the conditions under which the analysis is performed. As the bromine-addition figure of resin is 0.0 and that of linseed oil 102, this promised to be of some use; but it completely broke down on account of the changes made by the process of manufacture in the properties of oil and resin. Gladding's process for the estimation of common resin in mixtures with fatty acids, when tried on varnish, also failed to give satisfactory results. The author concludes that it is evident the processes used in oil analysis are not adapted to the analysis of varnishes. The oil and resin react upon one another in some way not understood, giving rise to new compounds, and it is necessary that something further should be known as to the nature of these before this analytical problem can be solved.

W. J. S.

The Viscosimetrical Examination of Butter. Dr. C. Killing. (*Zeit. für angewan. Chemie*, 1895, *Heft* 4, pp. 102, 103).—Since the publication of his former communications,* the author has examined a large number of viscosity apparatus, and has found that two may give identical times for water at 20°, without doing so for one and the same fat. The latter will only be similar when the body of the pipette, delivery tube, etc., are of exactly the same dimensions. From this it follows that the time taken by water to run out cannot be taken as a standard for fats, and, accordingly, the viscosity number, which depended on this relation, falls to the ground. This, however, makes no difference in the use of the method, but only in the manner of stating the results. Each apparatus, before being sent out, is now standardized for the mean "running out" time of butter as well as for that of margarine, and is thus ready for immediate use.

Since lard has occasionally been used to adulterate butter, the author gives the time taken by it in running out, and also that of beef fat. In an apparatus giving for butter the mean time of 3 min. 43.5 sec., and for margarine 4 min. 19 sec., the following results were obtained :

Lard.			Beef Fat.		
	min.	sec.		min.	sec.
1.	4	29.00	1.	4	35.50
2.	4	26.75	2.	4	33.50
3.	4	30.25	3.	4	31.00
4.	4	26.25			

As these both take a longer time than margarine, they would be detected even more readily than the latter.

C. A. M.

Note on the Adulteration of Commercial Peptones. M. L. Hugounenq. (*Journ de Pharm. et Chim.*, 1885, pp. 179, 180).—Two samples of peptone, examined by the author, gave an abundant reduction with Fehling's solution. The solution of the peptone, when warmed on the water-bath for an hour with a mixture of sodium acetate and phenylhydrazine chlorhydrate, deposited on cooling a magma of needle-shaped crystals, which, after purification by washing and recrystallization from boiling water, melted at 205°C. This melting-point of the osazone pointed to the

* See ANALYST xx., p. 66.

presence of ordinary glucose. But the absence of alcoholic fermentation in the suspected liquid, and the fact that the compound with phenylhydrazine, although closely resembling phenylglucosazone in the form of its crystals, its melting-point, and behaviour towards organic solvents, was yet distinguished by its solubility in boiling water, showed that the substance was probably lactose. This was confirmed by Rübner's reaction, in which, when a dilute solution of lactose is saturated with a hot neutral solution of lead acetate, and ammonia added drop by drop to the boiling solution, the liquid changes to yellow, then to orange, and finally to red. With pure peptone this does not occur, while in the two adulterated samples it did so very markedly. The amount of milk sugar estimated with Fehling's solution was found to be upwards of 40 per cent. in each case.

The Kjeldahl determination of the nitrogen was also very conclusive. A good peptone from the Lyons hospital yielded 12.72 per cent., while in the two adulterated peptones only 8.34 per cent. was found.

C. A. M.

Chemical Process for Recognizing Horse-flesh. M. Humbert. *Recueil de médecine vétérinaire* (through *Journ. de Pharm. et Chem.*, 1895, pp. 195-198).—In the year 1892 over 20,000 horses were slaughtered in Paris for the purpose of food. Much of their flesh was used in sausages, the vendors of which were compelled to indicate the nature of what they sold. It is thus a matter of great importance to be able to detect horseflesh when mixed with other kinds of meat. The Belgian chemists, Brautigam and Edelmann, have recently shown that it could only be confounded with the flesh of the human foetus and the foetus of animals. For its detection the reaction of iodine on broth obtained from the flesh is used both by these chemists and the author. The latter proceeds as follows: "About 50 grammes of the muscular tissue are cut into small pieces and boiled for an hour with 200 c.c. of water. After cooling, nitric acid is added in the proportion of 5 c.c. to 100 c.c. of broth, and the liquid filtered. To some of the filtrate in a test tube iodine water is added drop by drop, so as not to mix the liquids, and a reddish violet circle will show that horseflesh was present in the original sample. The iodine water is prepared by saturating hot water with iodine. When the reaction is doubtful, it is advisable to replace water by a 3 per cent. solution of KOH. The reaction is more pronounced when the iodine water has been recently prepared. Of ten samples of horseflesh obtained from different dealers in Paris, seven showed the colour very plainly, in two it was less pronounced but still clear, while in the last it was doubtful. In no case was there any coloration with beef, veal, mutton or pork. Beef broth left in contact with the iodine for ten days showed no signs of change. The flesh of the ass also gave a negative result, but with that of the mule the reaction was the same as with horseflesh. A mixture of equal parts of horseflesh, beef, veal, mutton and pork, showed the coloration, but it was less pronounced than with horseflesh alone.

C. A. M.

The Determination of Phosphoric Acid. H. Pemberton, Jun. (*Jour. Amer. Chem. Soc.*, 1895, xvii., pp. 178-181).—The author cites several precautions to be observed in using his method:

In standardizing the alkali, phenol-phthalein, *not* methyl orange, must be used as

the indicator. Since KOH, as bought, usually contains alumina, the neutral reaction is consequently shown with methyl orange before it appears with phenol-phthalein.

When precipitating the phosphoric acid with the aqueous solution of molybdate, the liquid must be removed from the flame as soon as the boiling point is reached, and *no more heat applied*. The temperature, on adding the molybdate, will then be about 70°, and no water-bath is required.

In order to obtain the precipitate in a granular state, the molybdate solution should be added very slowly from the pipette, the flow being interrupted by closing the end with the finger, and the solution stirred as it goes in.

The washing of the precipitate, which is a difficult matter, is best done on a Hirsch funnel or in a Gooch's crucible, with the aid of a suction pump. A precipitate containing about 50 milligrammes of phosphorus pentoxide is about the largest quantity it is advisable to work on, and where suction is not used, 30 to 40 milligrammes should be the limit. A precipitate containing 40 milligrammes is thoroughly washed on the Hirsch funnel by 300 c.c. of water. C. A. M.

CORRESPONDENCE.

COCOA-NUT OIL IN LARD COMPOUNDS.

To the Editors of THE ANALYST.

SIRS,—In the February issue of *THE ANALYST* I see that the question of cocoa-nut oil as a lard adulterant has again come up for discussion. As there seem to be very few recorded instances of the presence of this substance in lard compounds, the following example, which I came across a few years ago, may be of interest to your readers.

Dr. Wiley, in his exhaustive report on American lard (United States Department of Agriculture, Div. Chem., Bulletin 13), says: "It is probable that in this country lard is never adulterated with cocoa-nut oil for commercial purposes," and, as far as I know, there have been, so far, no cases reported from this continent.

The sample in question was taken in St. John's, New Brunswick, and was said to have been manufactured by Chase and Deeken, of New York. It was not sent to me until some four months had elapsed since its purchase, and when received it was in a rancid condition. The material was strikingly hard, and had a slightly yellowish colour. It contained 8.44 per cent. of water and 0.57-substances insoluble in ether. The filtered fat had a pale, lemon-yellow colour when cold, a melting-point between 46° and 47° C. and a specific gravity at 99.5° C. (water at 4° C.=1), 0.86449. With the Becchi silver test a walnut coloration was produced, but the reduction took place very slowly. The Hubl number came out 47.8 and the saponification equivalent 259. The crystals deposited from ethereal solution were unlike those from either pure lard or the ordinary lard compounds. Now, though many market samples of lard compound nearly reach the above specific gravity, and the iodine number of a genuine lard may perhaps be as low as 48, yet no possible mixture of lard, stearine, and cotton-seed oil could give both these numbers. I came to the conclusion that the mixture contained about one-third its weight of cocoa-nut oil and a moderate amount of cotton-seed oil.

It is, I believe, claimed that cocoa-nut oil may be so treated as to destroy its characteristic taste and odour. In this instance, though the taste was not apparent at first—perhaps owing to the rancid condition of the sample—the material left a persistent and unmistakable after-taste of cocoa-nut. I may add that it is contrary to my ordinary experience to find more than traces of water in American lard compounds, though I have sometimes found as much as 30 per cent.

EDGAR B. KENRICK.

St. John's College, Winnipeg,
March 6, 1895.

THE ANALYST.

MAY, 1895.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held on Wednesday evening, April 3, at the Chemical Society's Rooms, Burlington House. In the absence of the President, Mr. Otto Hehner took the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election as members of the Society: Mr. H. Irving Foster, The Elms, Anlaby Lane, Hull, formerly assistant, and now partner, of Mr J. Baynes; and Mr. Alexander Poole Wilson, Analytical Chemist, Knocklong, co. Limerick.

Papers were read on the following subjects:

"The Composition of some English Cheeses." By W. Chattaway, T. H. Pearmain, and C. G. Moor, M.A.

"The Testing of Oils by the Oleo-refractometer." By T. H. Pearmain.

"Some Errors in Commercial Weighing." By S. Rideal, D.Sc.

The publication of these is unavoidably held over.

THE INTERPRETATION OF THE RESULTS OBTAINED UPON THE CHEMICAL AND BACTERIOLOGICAL EXAMINATION OF POTABLE WATERS.

JOHN C. THRESH, D.Sc., M.B., D.P.H., F.I.C.

Medical Officer of Health to the Essex County Council.

(Continued from page 91.)

The microbes found in water are chiefly bacilli. Micrococci are comparatively rare, whilst spirilla are not uncommon, especially in polluted waters. Already over two hundred distinct species of microbe have been discovered in potable waters, and amongst these are several which are pathogenic, or disease-producing. According to Professor Percy Frankland,* these are:

Typhoid bacillus.
Cholera spirillum, or "comma
bacillus."
Tetanus bacillus.
Anthrax "
Tubercle "
Bacillus brevis.
" capsulatus.

Bacillus proteus fluorescens.
" coli communis.
" hydrophilus fuscus.
" pyocyaneus.
Staphylococcus pyogenes aureus, and
the organisms causing septicæmia
in mice and rabbits.

* *Journal of State Medicine*, January, 1894: "The Bacteriological Examination of Water."

Up to the present, however, the only diseases which are known to be caused by drinking specifically-infected water, and the micro-organisms of which have been with certainty discovered in such waters, are cholera and typhoid fever. Doubtless, further research will add to this short list, but as yet the organisms causing malaria, dysentery, and other diseases, believed to be produced by specific microbes entering the system with the drinking water, have not been actually identified therein. The utmost, therefore, that can be expected of the bacteriologist is that he should discover and identify the cholera or typhoid bacillus, should either of these organisms be present in a sample of water submitted to him for examination. The multitude of other bacilli present, however, renders this a difficult and often impossible task; the search has been likened to the finding of a needle in a stack of hay. Whilst, therefore, the absolute identification of the specific cause of cholera or typhoid fever establishes its presence, the failure to isolate it is no proof of its absence. As a matter of fact, numerous samples of water, credited with the production of one or other of these diseases, have been examined with negative results. As examples may be quoted the examinations of the water supplies to Hamburg and Altona during the cholera epidemic, and the water supplies to Worthing, and to the towns in the Tees valley, during the outbreaks of typhoid fever, which recently occurred there. Although the Elbe was known to be polluted with cholera excreta, the comma bacillus was never discovered in the imperfectly-filtered river water, to the use of which Koch and others, who investigated the outbreaks, attributed their occurrence. At the commencement of the second serious epidemic of typhoid fever at Worthing, two samples of the water were submitted to bacteriological examination by Professor Crookshank. He found that they contained far fewer bacteria than the water supplied to King's College, and that there was a marked absence of liquefying colonies. "There was no colony of typhoid fever bacilli, and no bacillus to which suspicion could be attached of producing typhoid fever." He concluded, from the results of his bacteriological examination, "that both samples of the Worthing water rank as very pure water." Considering that during the construction of additional works in the spring, a fissure was opened which discharged into the wells a large volume of water, polluted by surface drainage, and leakage from defective sewers, and that this mixture of well and surface water thereafter was supplied to the town, and was the water examined by Dr. Crookshank, it is not surprising that the results of these and other examinations were considered by the public as "most remarkable." Chemical examinations made from time to time also failed to detect any pollution. The following statements, made by the Deputy Mayor of Worthing* at a meeting of the Town Council, held July 18, 1893, are particularly interesting, not only as showing how little reliance can be placed upon either the bacteriological or chemical examination of drinking waters, but also as showing the disastrous results which may follow misplaced confidence in these results. The Deputy Mayor, at the above meeting, after speaking of the finding, about two months ago, of the fissure which gave to the town an enormous additional yield of water, said: "We congratulated

* From Report in the *Sussex Coast Mercury*, July 22, 1893. Worthing has a population of about 17,000, and during the year 1893 nearly 1,500 cases of typhoid fever occurred. (It is only fair to add that previous samples of water from the same source had been condemned by Professor Crookshank as showing unquestionable evidence of pollution by filth.)

ourselves upon that fissure, but I think there is no doubt, and certainly no member of the Sanitary Committee has any doubt, that it is to that very fissure the whole of the difficulty we are sustaining, and have sustained, is entirely due." He then referred to the various chemical and bacteriological analyses which had been made, resulting in the water being pronounced thoroughly good and pure. Notwithstanding these results, the committee cautioned the public that they should boil the water, and the boiling went on until the first outbreak practically ceased. "We were hoping," he said, "that the difficulty had ceased, and that we were to have no more typhoid among us; but, unfortunately, another analysis was made by Dr. Crookshank, the water being taken from two or three different sources, and each sample was declared to be good. 'Perfectly pure' were, I think, the doctor's words. Well now, to that, I am afraid, to some extent, we may attribute the cause of the second outbreak. It was stated publicly, with the best intentions, to allay public excitement and the panic which was prevailing, that the water was perfectly pure, because we had the best evidence that it was so; and I have no doubt that the public, who do not like the trouble of boiling every drop of water they drink, ceased the boiling, and thus the second outbreak came upon us, and is still going on." It is quite unnecessary to point the moral of this plain statement of facts. As it has been found impossible to dam out the water from the prolific, but fatal fissure, the present source of supply is being abandoned. A proposal to attempt the purification of the water by filtration through sand has not been acted upon, Dr. Thorne having brought under the notice of the Sanitary Authority Professor Koch's experience, to the effect that, "even under favourable circumstances, sand filtration cannot give absolute protection against the danger of infection." During the Tees valley epidemic also the water was repeatedly examined bacteriologically. Although an excessive number of micro-organisms was found, sufficient, in fact, to justify the opinion that the water was polluted, the typhoid bacillus was not once discovered.

It has recently been pointed out that the so-called typhoid bacillus (Eberth's) is often absent from typhoid stools, and that the *bacillus coli communis*, which is invariably found in all stools, is capable under certain conditions (probably by growth in cesspools and sewers) of acquiring pathogenic properties in man. It is even, by many, believed that this is either a degenerate form of Eberth's bacillus, or that it is capable of taking on the same properties, and of causing the same disease—typhoid fever. Such being the case, all waters faecally polluted may be capable of producing this disease when all the circumstances are favourable, and, therefore, must be looked upon with the gravest suspicion, whatever the results of bacteriological or chemical analyses.

All surface waters contain large numbers of micro-organisms, but freshly-drawn deep-well waters, and waters from deep-seated springs, are almost sterile. When such pure waters are kept for a few days, however, the number of micro-organisms increases enormously. Professor P. Frankland says that such a water, containing only, say, five microbes per cubic centimetre when freshly drawn, may, even if kept in a sterile flask and protected from aërial contamination, contain, after a few days, perhaps 500,000 in the same volume, or, in other words, as many as are found in slightly-diluted sewage. He points out, however, that whilst in sewage the numbers

only gradually diminish, in these pure waters, "after the rapid increase in numbers; follows a correspondingly rapid decline, so that the numbers again very soon fall below those found in impurer surface waters." It follows, therefore, that the purest water which has been kept a few days may be confounded with a water from the filthiest source, and that even if the number of micro-organisms found in a water is to be taken as a criterion of its purity or otherwise, the bacteriological examination must be made before such multiplication can have ensued. In freshly-drawn deep-well and spring waters there should be few or no bacteria; in the purest mountain streams and lakes there should not be more than a few hundreds in a cubic centimetre (15 drops). In ordinary river waters from 1,000 to 100,000 may be found in the same volume, whilst in sewage there may be several million. Rain, hail, snow, and ice are not free from bacteria, though usually the number contained therein is small.

In 1887 Professor W. R. Smith made a series of experiments for the Local Government Board (*vide* Report of the Medical Officer, 1887) on the differentiation and identification of micro-organisms found in water supplies. The results gave evidence of the multifarious character of the organisms in question, and illustrated the need for caution against drawing general conclusions from the results of cultivating water organisms by any single method. In the same year Dr. Dupré, F.R.S., reported to the Board on changes effected in the aeration of certain waters by the life processes of particular micro-organisms under different conditions of temperature, light, and nutrient material, but the results obtained seem of no practical value. "The process of oxygen consumption was found, as might be expected, to be influenced by these circumstances, but it would not yet be safe to formulate general inferences from the facts."

Koch, in an able article on Water Filtration and Cholera,* has endeavoured to set up a standard of purity based upon the number of bacteria, capable of cultivation in certain media, contained in a given quantity of the water. He would regard even filtered river water containing over 100 micro-organisms in a cubic centimetre as open to suspicion; but, as we have just seen, he does not regard such water, if once polluted, as absolutely safe, however careful and thorough the filtration; but to this question we shall have shortly to refer again. The Royal Commissioners on Metropolitan Water Supply do not entirely concur with this conclusion. They point out that the typhoid bacillus is, so far as is known, only found in human excrement, and that it has not yet been found to retain its vitality when in faecal matter for more than fifteen days; that in all ordinary waters there exist organisms which "undoubtedly exert an influence in diminishing the vitality of the typhoid bacillus; that exposure to direct sunlight destroys these bacteria; that they have a tendency to subside more or less rapidly in all slowly-moving waters, and to be carried down with other matters held in suspension; and that there are strong grounds for believing that small doses either of cholera or of typhoid poison may be swallowed with impunity. Such being the case, they fall back upon the "evidence of experience," and whilst acknowledging that the various water supplies to London are contaminated with sewage, which may, and often does, contain the specific poison of typhoid fever, and may contain the bacillus of Asiatic cholera, they "state without hesitation,

* Translated by J. A. Ball, Esq., and published by the Local Government Board.

that, as regards the diseases in question, which are the only ones known to be disseminated by water, there is no evidence that the water supplied to the consumers in London by the companies is not perfectly wholesome." In other words, these polluted river waters, which have undergone a filtration far less perfect than that required by Koch, since London water usually contains many hundreds of micro-organisms in the cubic centimetre, are perfectly safe and wholesome.

Many bacteriologists now accept the number of micro-organisms present in a water as the measure of its pollution, and some have not hesitated to approve or condemn waters according as they contained few or many organisms, even where the results of chemical analyses directly contradicted their conclusions.

The attempt to set up a standard of purity based upon the number of micro-organisms in a given quantity, is as illogical as the old chemical standards. Both depend upon quantity, whilst the real point at issue is the quality. In reputedly good waters it has been observed that the micro-organisms present capable of liquefying gelatin by their growth are few in number, whilst in sewage-polluted waters they abound; but this fact is of little value, since it only enables somewhat gross pollution to be detected, and most of these liquefying organisms are perfectly harmless. Bacteriology, like chemistry, may tell us something of hazard and impurity, but neither can be depended upon to determine with certainty whether a water is actually injurious to health. To condemn one water because it yields a little more albuminoid ammonia than another, or because it contains a few more organisms than another, when we know nothing of the nature of the substance yielding the ammonia, and nothing of the character of the organisms, is obviously so illogical as to be absurd, and yet this is what is almost invariably done. Bacteriological, microscopical, and chemical examinations must always be associated with a thorough investigation of the source of the water, to ascertain the possibility of contamination, continuous or intermittent. Then, and then only, if everything be satisfactory, we may be justified in speaking of safety and of freedom from risk; but where either the bacteriological, microscopical, or chemical examination is unsatisfactory, the inquiry into the history of the water must be most careful and complete, and a guardedly-expressed opinion given only after a full consideration of the bearing of the one upon the other. The possibility of accidental pollution is a point too often overlooked; yet it is to such accidental pollution that outbreaks of disease are most frequently attributed, and of this the examination of samples of water prior to the occurrence of the contamination may tell us little or nothing. The danger of such pollution does not, unfortunately, vary with the amount of any constituent found in the water, and a source yielding a water of great chemical and bacterial purity may be more liable to occasional fouling than a source yielding water containing excessive quantities of chlorides and nitrates, or even of unoxidized organic matter, or large numbers of living organisms.

Although a mere analysis cannot guarantee us purity and safety, yet it very frequently can reveal to us impurity and risk. When the source of a water, upon most careful examination by an expert, is found to be free from all danger of pollution, and the chemical examination proves that the inorganic constituents are unobjectionable both in quantity and quality, and that organic matter is absent or present in barely appreciable amount, then safety, so far as human foresight can be trusted,

may be guaranteed. If organic matter be present in appreciable quantity—that is, if the water yield such a quantity of organic nitrogen and carbon, or albuminoid ammonia, or requires such an amount of permanganate for oxidation as to render it of suspicious or of doubtful purity—a study of the history of the water and of its geological source may, and generally does, enable an opinion to be formed as to the nature of the organic matter, and as to whether it is of an innocuous or dangerous character. Chemical analysis, therefore, has its use; it is only when it is made the sole arbiter between safety and risk that it is abused, and is liable to lead to errors fraught with most disastrous consequences. Let the analysis be as careful and complete as possible, but let the results always be interpreted in the light afforded by a searching examination of the source of the sample. Let all so-called standards be abandoned as absurd, and let the opinion as to whether water is dangerous or safe be based upon a full consideration of other and more important factors.

On the opposite page are arranged in a tabular form the results of the chemical analysis of a number of waters which are reputed to have caused serious outbreaks of typhoid fever, yet were passed by analysts of repute as good potable waters. A few other analyses are also inserted to exemplify points discussed in the paper. The following remarks bearing upon each of the analyses supplement the table.

Remarks.

1. Analysis of water from the river Ouse below where it receives the sewage of Buckingham. Examined for the Town Council, February 29, 1888, by W. W. Fisher, Public Analyst. Report: "Does not appear from the analysis to contain sewage matters." Quoted by Dr. Parsons in his report to Local Government Board on an outbreak of enteric fever in 1888, as a "further illustration of the inability of a chemist to prove the quality of organic matter in water when its quantity is small."

2. Analysis of the Buckingham public water supply by Mr. Fisher. Certified by him to be a first-class water, yet believed by Dr. Parsons to have been the cause of the above outbreak.

3. Analysis of the Beverley water supply from borings in the chalk by Mr. Baynes, July 18, 1884. In 1884 an outbreak of typhoid fever occurred here, and was investigated for the Local Government Board by Dr. Page. The evidence led him to conclude that the specific contamination of the water supply was the immediate cause of the outbreak. The water had been repeatedly analysed, and the analysis given was made "on the very border of the period when the water was acting as the epidemic agent." It was certified to be "of a very high degree of purity, and eminently suitable for drinking and domestic purposes." Specifically infected sewage from an asylum had been spread upon land near the well and reservoir.

4, 5. Analyses of water from the much-polluted Trent at (4) Torksey, and (5) Knaith, by Dr. Tidy, December 20, 1890. The analyst reported that "there is no evidence of the product of sewage contamination." From Dr. Bruce Low's report to the Local Government Board on the occurrence of enteric fever amongst the population using the Trent water, 1893.

6. Analysis of the well water supplying Houghton-le-Spring, April 24, 1889. Early in the month a sudden outbreak of typhoid fever occurred here, and a sample

NUMBER.	SOURCE OF LOCALITY.	APPEARANCE, ETC.	RESULTS IN GRAINS PER GALLON.							IN PARTS PER MILLION.				
			Total Solids.	Effect of Ignition.	Nitrite Nitrogen.	Chlorine.	Temporary Hardness.	Total Hardness.	Organic Carbon.	Organic Nitrogen.	Free Ammonia.	Alumina.	Nitrites.	Oxygen used in four hours.
1.	R. Ouse, Buckingham	Turbid and slight weedy odour	22.0014	1.213	.30	Trace	1.30	
2.	Ditto	Bluish tint, good in colour	37.507	1.109	.07	0	0	
3.	Beverley Water Supply	...	26.0	1.55	19.9	25.600	.01	
4.	R. Trent, at Tortsey	Turbid	26.4177	2.23	12.7	30.7	.12	.02	.07	...	1.34	
5.	" Korath	...	26.6177	2.23	11.2	30.8	.12	.02	.09	...	1.29	
6	Houghton-le-Spring supply	Colourless and nearly clear	34.4	2.100	.0313	
7.	R. Tees water, Middlesborough Supply, } Aug., 1890	Pale brown, turbid, peaty taste	7.300	.49	0	3.9	.70	.047	.00	
8.	R. Tees water, Middlesborough Supply, } Oct. 22, 1890	Very slightly turbid, peaty taste	9.100	.56	1.9	6.1	.80	.010	.00	
9.	R. Tees water, Middlesborough Supply, } Oct. 27, 1890	Cl-ar, dark yellow	10.800	.5002	.12	0	...	
10.	R. Tees water, Middlesborough Supply, } October 29, 1890	Light brownish yellow	12.07001	.12	
11.	R. Tees water, Darlington Supply, } Dec. 2, 1890	Greenish yellow, clear and bright	10.5028	.700	.04	...	3.0	
12.	R. Tees water, Darlington Supply, } Jan. 1, 1891	Clear, no peaty taste	13.4036	.81	4.8	8.2	.08	.020	.00	
13.	R. Tees Water, Darlington Supply, } Feb. 9, 1891	Brownish yellow, not quite clear	7.2011	.3503	.03	...	3.27	
14.	R. Tees water, Stockton Supply, Aug., } 1891	Dark brownish yellow, almost opaque	8.5400	.494904	...	10.4	
15.	R. Tees water, above Barnard Castle, 1887	...	9.38354	4.16	
16.	" below ditto (Darlington), 1887	...	7.7	Blackened	Trace	.6500	.014	0	4.01	
17.	Mountain Ash Water Supply	Very pale brownish	6.2025	.16	1.5	5.0013	.03331	
18.	Massachusetts, Unpolluted Surface Water	Slightly turbid	3.6020	.58002	.020	
19.	Polluted ditto	"	5.0010	.09017	.12	
20.	Merrimack River	"	2.4001	.09002	.10	
21.	Chicopee River	"	2.804014	.24	
22.	Boston Supply. Purest reservoir	"	2.7004	.1621	.24	
23.	" Boston Supply. Mystic lake	"	7.4	Slightly charred	.04	1.3	...	4.120	.05	0	.75	
24.	Essex. Deep well	"	95.013	25.6	4.0	4.001	.24	0	3.25	
25.	" Same well, a few weeks later...	Blackened	113.0	...	1.44	14.8	17.0	19.002	.08	0	1.75	
26.	" Shallow well, near surface	Brownish	94.0	...	4.14	16.1	13.5	35.002	.12	Trace	2.50	
27.	" Same well, near bottom	Charred	119.0	...	4.33	22.0	17.3	45.520	.0943	
28.	Well at Melton Asylum, Oct. 11, 1893	Turbid and yellow	42.	...	0	12.729	.2433	
29	" " " "	...	45.5	...	Trace	6.421	.2062	

of the water was at once sent for analysis. The analyst reported: "This water is very free from indication of organic impurity. . . . It is a good water for drinking purposes." Dr. Page, who investigated this outbreak for the Local Government Board, found that sewage from a farm three-quarters of a mile away was discharging into the well at a point 45 feet from the surface.

7-14 form a very interesting series of analyses by chemists of the highest repute, of the Tees water as supplied to the towns in the Tees Valley. Two outbreaks of enteric fever occurred in these towns, the first between September 7 and October 18, 1890, and the second between December 28, 1890, and February 7, 1891. Dr. Barry reported upon them to the Local Government Board. He found the river above the intake of the water companies excessively polluted by sewage, cesspool drainage, etc. It is with reference to the relation of this water to the typhoid epidemics that Dr. Thorne says: "Seldom, if ever, has the proof of the relation of the use of the water so befouled to wholesale occurrence of typhoid fever been more obvious or patent." The analyses now quoted were made before, during, and after the epidemic periods, yet, as will be seen, in not a single instance did the chemical examination indicate either pollution or danger.

7. Analysis of the Middlesborough water supply by Dr. Frankland, F.R.S., August 23, 1890. Report: "Peaty . . . but in all other respects the water is of excellent quality for domestic use, and it is free from any trace of sewage contamination."

8. Ditto, October 23, 1890. Report: "With the exception of a peaty taste, it is in all respects of excellent quality for dietetic and all other domestic purposes."

9. Analysis of the Middlesborough water supply by A. H. Allen, F.I.C., October 27, 1890. Report: The results "negative any suspicion of contamination of sewage or cesspool drainage. . . . No suspicious results were obtained on bacteriological and other microscopical examination."

10. Analysis of the Middlesborough water supply by Messrs. Pattinson and Stead, October 29, 1890. Report: "Perfectly wholesome and free from any sewage contamination. . . . The microscope reveals nothing of an objectionable character."

11. Analysis of the Darlington water supply by F. K. Stock, County Analyst, December 2, 1890. Report: "I have no hesitation in saying that the Tees water as at present being supplied to consumers is of good and wholesome quality."

12. Analysis of the Middlesborough water supply by Dr. Frankland, F.R.S., January 1, 1891. Report: "Of excellent quality for dietetic and all domestic purposes."

13. Analysis of Darlington water supply by W. F. K. Stock, County Analyst, February 9, 1891: "I am of opinion that the Tees water as supplied to the town on January 29, 1891 (the date when the sample was taken), was good and wholesome drinking water."

14. Analysis of the Stockton water supply by A. C. Wilson, Borough Analyst, August, 1891. Report: "Heavily charged with organic matter of vegetable origin. There is, however, no appearance of animal pollution."

That the river Tees some miles above the company's intake is grossly polluted

with sewage no one has denied, yet these waters, upon analysis, were said to be pure and wholesome, and free from any trace of sewage contamination. As they are stated by the most competent authorities to have been the cause of the extensive epidemics of typhoid fever, most of them must have been absolutely poisonous at the time they were examined.

15, 16. In 1887, when an inquiry was being held to investigate the pollution of the river Tees, the late Professor Tidy examined a number of samples of water therefrom. No. 15 is the mean of several analyses of samples taken above where the river receives the sewage of Barnard Castle, and No. 16 is the mean of several analyses of samples taken at Darlington, fifteen miles below Barnard Castle. Notwithstanding the sewage poured in at this town, and at points nearer Darlington, Dr. Tidy reported that the water at the latter place was rather better than at the former, and was good and wholesome. He adds: "I am of opinion that if the quantity of sewage discharged into the river at Barnard Castle was enormously greater than at present, the self-purifying action of the water would be amply sufficient to oxidise every trace of sewage impurity within a short distance of the outfall. Further, I am of opinion that Darlington would not be prejudiced (although the river is the source of the water supply) even if an outbreak of fever or cholera were to occur at Barnard Castle."

17. Mean of four analyses of the Mountain Ash water supply (spring and surface water) by Dr. Dupré, November, 1887. A serious outbreak of typhoid fever occurred here, commencing in July, 1887, and continuing until October. Mr. John Spear investigated it for the Local Government Board, and attributed the epidemic to insuccion of filth into one of the water mains during intermission of the service. Dr. Dupré found the samples almost identical from a chemical point of view, and very pure and free from any indication of sewage pollution. The two samples, however which were taken from the taps, after six hours' intermission, were found, when examined *microscopically*, to contain fungoid growths and large animalculæ, which were absent from the two other samples.

18-23 are analyses quoted from the *Report of the Massachusetts State Board of Health*, 1890-92.

18. A sample of unpolluted surface water containing more nitrates and yielding more albuminoid ammonia than (19) a sample of surface water known to be polluted by sewage.

20. The average of a series of monthly examinations of the water of the Merrimack River, supplying the town of Lowell during 1891, when typhoid fever was epidemic, and attributed to the water being specifically infected nine miles above the intake.

21. Analysis of water from the Chicopee River, supplying the city of Chicopee. Specific pollution is believed to have taken place seven miles above the intake, and to have caused an outbreak of typhoid fever in the city.

22. Analysis of the water from No. 4 reservoir, the purest of the four water supplies to the city of Boston, and (23) of the water from Mystic Lake, the most impure supply, showing that the albuminoid ammonia yielded by the latter does not exceed that yielded by the former.

24, 25 are waters from a deep well in Essex ; (24) collected during dry weather ; (25) collected eighteen hours after very heavy rain. This well water is liable to most serious pollution, yet a report based merely upon the results of the first analysis would most certainly have been favourable.

26, 27 are waters taken by me from the same well ; 26 from near the surface, and 27 from near the bottom.

28, 29, 30. Water from a carefully-constructed bored well, which is believed by Dr. George Turner to have been the cause of a fatal outbreak of dysentery. The variation in character led Dr. Turner to suspect that surface water was being pumped from the bore-tube, and upon pouring a solution of lithium chloride into a hole dug by the side of the tube, lithium was soon afterwards found in the water drawn from the tube.

DISCUSSION.

Mr. HEHNER said he thought Dr. Dupré's paper came not a moment too soon. However great the benefits of that branch of science had been in other divisions of knowledge, far too much importance had been attached to bacteriology in connection with the examination of water. When bacteriology was first introduced into water-analysis, a mere counting of the number of organisms was thought to be sufficient, without taking into account the nature of the organisms themselves. This position was soon shown to be obviously untenable. A distinction was then made between organisms liquefying gelatin and those devoid of that property. This distinction also broke down ; many harmless bacteria liquefy gelatin, while many pathogenic bacteria fail to liquefy this medium. At the present time work was directed into more scientific channels, but a point had hardly been reached when definite results were readily obtainable. No doubt, in many cases, after outbreaks of disease, definite pathogenic organisms had been discovered in certain water-supplies, but bacteriology in such cases came after the event. The chemist had taken a perfectly distinct attitude ; when his analytical data indicated sewage pollution, he had warned against the use of the supply, sewage being potentially dangerous, not necessarily injurious. Thousands of cases could be cited in which chemistry had thus been of the greatest value, while he had yet to hear of the first case in which bacteriology, as applied to water-examination, had done similar service. Chemists certainly could not say, upon their most definite results, that any sample they might examine was actually injurious to health ; but surely modern sanitation did not require such proof, when the presence of danger, in the form of pollution, was established. If bacteriology could be applied so as to assist the chemist in this respect it would be most heartily welcomed, but at the present time it did not appear to be sufficiently advanced to do this. Even in fluids where pathogenic bacteria were bound to be present, most careful search had not unfrequently failed to pick them out from the countless numbers of harmless ones. Thus Mr. Laws, in his recent report to the London County Council, showed that he had to go to the sewers quite close to fever hospitals before he succeeded in discovering in London sewage any typhoid bacilli. If bacteriology was to be taken as the sole guide, it would follow from this report of Mr. Laws, as also from a previous one, that the best air to breathe in London was that of the sewer, and the

safest liquid to drink was sewage itself; propositions so obviously absurd and contradictory to established experience that they needed no refutation.

Although bacteriology was thus at the present time incapable of materially assisting the chemist, it had yet done a great deal of harm, and undeserved discredit had been thrown upon chemical analysis upon the strength of a few exceptional cases of pollution where typhoid excreta had been directly introduced into water-supplies. While sound knowledge on the subject of the chemistry of water could only be obtained by years of work and experience, it appeared very seductive, by going through a two or three months' course of bacteriological study, to be able to become a master of the great problem:

He was quite in accord with Drs. Dupré and Thresh as to the fallacy of deciding from the result of a single determination. Nothing short of a very exhaustive analysis, and of a knowledge of the nature and composition of the natural unpolluted water of the district from which a given sample was derived, could enable the chemist to form a safe opinion in most cases. Dr. Thresh had most usefully pointed this out once more, although public analysts had been long agreed upon this point. The warning rather applied to medical men, who were much more apt to judge by arbitrary limits than analysts were.

Dr. GEORGE TURNER agreed with Dr. Thresh that it was not in the province of a medical officer of health to make analyses, and that such work ought not to be put upon him. It was, beyond doubt, better that it should be done by a chemist, who, having received a special training, would be more likely to do it satisfactorily. His opinion with regard to bacteriology was that it would one day take a very important part in the examination of water for drinking purposes, although it had been severely criticised. He was, however, also of opinion that although an analyst might be able to condemn a water upon his analysis only, it was most undesirable that he should give an opinion that a sample of water was fit to drink or safe upon the same grounds alone, without knowledge as to the presence or absence of possible sources of pollution, and the average composition of the water taken in the locality from the same source.

Dr. J. A. VOELCKER said that a chemist could not go to the extreme of pronouncing any water either absolutely injurious or absolutely safe. If a water were found to give evidence of the presence of sewage in it, it was perfectly justifiable to say that it might at any time become dangerous to health, but he did not think that it would be wise to go further than this. Nobody could doubt that the time would come when bacteriology would become of great assistance in the examination of waters, but hitherto it had been taken up in a very uncertain and dogmatic way, and chemists could not accept it as at present put forward. More definite information was needed as to the nature of the micro-organisms, and as to their behaviour, in order to enable bacteriology to rank as a reliable agent in the examination of water. The comparison of the sample under examination with another from the same source, of which the history was known, was very important, and bore upon what Dr. Dupré had said as to the impossibility of judging correctly by fixed standards, or from the determination of single constituents only. Numerous determinations were necessary, and it was only by a consideration of these in conjunction one with the other that a fair conclusion could be arrived at. It struck him that some public analysts could not have

borne this in mind in undertaking water analyses at the very low fees which they sometimes charged.

Mr. S. F. BURFORD said that he was of opinion that the consideration of any particular water should be governed by the character of the district from which it came, and if it differed to any considerable extent, even in only one particular of importance, from the typical water of the district, its condemnation was justifiable. He had lately analysed several samples of water whose only abnormality was a large amount of chlorine. In one case, a network of drains was situated near the source of the water, and as the typical water of the district contained only a small quantity of chlorine, he felt justified in condemning the sample.

Mr. CASSAL said that the work of water examination should only be undertaken by those who were specially qualified by education, training, and experience. Chemical and bacteriological examinations should only be made by men who were specialists in those branches of knowledge. He held that it was a mistake for medical officers of health to practise analysis, however well qualified some of them might be to undertake that work. Their doing so injured their own profession. Public authorities in some cases forced, or tried to force, their medical officers to undertake analytical work—a course of action which was detrimental to both the medical and chemical profession, as well as to the public.

With regard to the misleading way in which the results of water analyses were often interpreted, the public themselves were to blame for wilfully keeping analysts in the dark as to the source and nature of the supplies from which samples had been taken for examination. It was true that many analytical chemists acted very indiscreetly in regard to the condemnation or passing of samples of water, but so also did many medical men who practised analysis. Absolute safety with respect to a water-supply was a thing that could never be asserted on the strength of chemical or bacteriological examinations, or both combined, not even with the additional information that might be afforded by inspection of sources of supply. A great deal too much had been made of the value of bacteriological methods. Whatever might be thought of a person who passed a water as perfectly safe upon the results of chemical analysis alone, the bacteriologist who ventured to pass a water as safe upon negative bacterioscopic results was a most dangerous enthusiast. Bacterioscopic methods at present afforded nothing more than a test of the roughest and most misleading description for the existence of pollution, and he (Mr. Cassal) would venture to say that by their very nature those methods must of necessity fail to give indications of any great practical value.

Dr. RIDEAL said that it was very useful to know the total nitrogen present in a water, and this could be determined without much labour by the Kjeldahl method. Besides indicating animal pollution, it would also throw light on any toxic poisons that were present in appreciable quantity, and these may be present after the bacteria giving rise to them have disappeared. The phosphoric acid determination had not been referred to, but in some cases it was of great use.

When bacteriology was sufficiently developed to enable the character and nature of the micro-organisms to be identified with certainty, it would be a very valuable assistant, but merely counting the number of organisms was worse than useless.

Dr. DUPRÉ said he had really nothing to answer. He cordially agreed with much contained in Dr. Thresh's paper. It was essential that the chemist should be told everything that was known about the water he had to examine. He himself frequently had had the advantage of working in conjunction with a sanitary engineer, and the results of their joint labours were very satisfactory—far more so than would have been obtained had they worked separately—showing clearly how important it was, in forming an opinion, to be in possession of complete data of all kinds. He was perfectly convinced that no arbitrary standards were possible. One should work by the general standard of the district, as suggested years ago by Mr. Hehner and himself. He pointed out that in stating the results of an examination it was advisable to adhere to one method, as any alteration—such as changing from grains per gallon to parts per 100, or per 1,000, or even, as he had seen, to parts per 1,000,000—had the effect of apparently altering the proportions of different constituents, rendering the analysis at first sight very misleading.

Although absolute safety could not be guaranteed, it was possible, by employing analysis in conjunction with inspection by sanitary officers, to guard against regular and constant pollution, though accidental pollution might occur, and could not be guarded against by any method of examination.

One reason why the results of bacteriological examinations were less reliable was that suspended matters, and especially living organisms, were never so evenly distributed through the water as were the matters in solution dealt with by the chemist, so that a fair sample of any dangerous matters was more likely to be obtained.

The PRESIDENT said that a great deal of acumen and experience were needed in properly interpreting the results of a water analysis. The time had long gone by when the determination of one or two constituents was considered sufficient to enable an opinion as to purity to be formed.

He thought that at present chemistry could say more that was really useful than bacteriology could, although it was probable that in the not very distant future a great deal would be learnt from bacteriology. He did not despise bacteriology, but when from the report of Messrs. Laws and Andrewes it was learned how little of the typhoid bacillus could be isolated from London sewage, he thought it must be concluded that at present bacteriology did not afford so much help as chemistry in the examination of waters.

The following remarks have been received from Mr. W. W. FISHER :

As I was unable to stay for Dr. Thresh's paper, and had not the advantage of hearing it and taking part in the discussion, I should like to offer a few criticisms on the line of argument by which he reaches the conclusion that in most cases the chemical examination of a water is quite useless. I do not believe the inference is valid, that because an analysis is unable to tell you everything about a water such information as it affords is worthless. Every chemist having the responsibility of giving an opinion upon the character of drinking waters must be alive to the fact that an error in judgment may lead to the gravest consequences, either in regard to the life and health of the persons using it, or in respect of hardships and expenditure which

may be caused by groundless condemnation of a water supply, and will therefore agree with the view that all possible information about a water should be obtained. Where important facts as to the origin of a water are not given, they may generally be obtained for the asking; while if from ignorance or other reasons information is refused, surely a chemist is entitled as much as any other professional man to decline to give his opinion on inadequate data.

Dr. Thresh urges on us that "mere chemical analysis is powerless to prove that any water is of such a quality as to be incapable of producing disease"—a statement which has long been in active service in a highly-official capacity. But what more does it amount to than an assertion that analysis cannot prove a negative? What conceivable methods could ever or will ever *prove* a given water incapable of producing disease? But if the assertion means only that after adequate chemical examination there still remains a residuum of doubt and risk, the question is narrowed to one of reasonable probability. I claim that experience has abundantly shown that chemical methods are capable of classifying waters, selecting those which are fit for drinking, and rejecting those unfit for the substantial reason that the use of polluted waters involves manifest risk and danger to health. We are told that chemists can tell of risk, but not of safety (Sir G. Buchanan, Royal Commission on the Metropolitan Water Supply, 1894), which is a mere truism, as the desired safety is but the absence of risk. Imagine a board of railway directors (if I may venture on the supposition) taking the view that as an engineer can only tell of risk and not of safety, any examination of their bridges was worthless, as it could not "prove the bridge incapable" of breaking, or, to use Dr. Thresh's own expression, he will "never be justified in certifying that it can be used absolutely without risk." The answer to this seems to be that there will always be cases of engineering accident, and equally of water pollution from unforeseen causes; but such occurrences will be more frequent if the more important methods of safeguard are disparaged and despised because they do not cover the whole field of inquiry.

No waters have been more thoroughly and continuously examined than the supplies of the London Water Companies; they have been violently attacked on *a priori* alarmist grounds, and strenuously defended on the results of chemical analysis, and yet, notwithstanding the myriads of microbes and the brigades of bacteria shown to inhabit every gallon of the river water, a Royal Commission had reported clearly and unmistakably in their favour. In reference to the bacteriological aspect of the question, they say that "as regards the diseases typhoid and cholera, which are the only ones known to be disseminated by water, there is no evidence that the water supplied to consumers in London is not perfectly wholesome"; and they further add: "We are strongly of opinion that the water as supplied to the consumer in London is of a very high standard of excellence and purity." These weighty conclusions are the outcome of a long and thorough inquiry; and the most important part of the evidence on which they are founded is the chemical evidence of the standard of purity, which is only confirmed and strengthened by the biological evidence, and the experience of the responsible health officers for London. Dr. Thresh, however, would set this all aside as of trifling importance compared with the results obtained from inspection and knowledge of the history of the water. Infor-

mation as to the results of inspection and the history of the waters was indeed abundantly supplied, some of the most alarming character highly coloured with individual prejudice; but such information could not outweigh the impersonal irrefragable results obtained by scientific chemical methods. After the experience we have gained during the past thirty years, I maintain that the chemical examination of water stands in a stronger and more assured position than ever, and gives information of the highest value for judging of the quality of potable water.

NOTE ON UNUSUAL SPECIMENS OF MILK.

By SIR CHARLES A. CAMERON.

It seems a little hard on a vendor of milk which contains more than 4 per cent. of fats, to certify that it was largely adulterated with water. I have had frequently to do so. The latest case of the kind occurred during the present week. Sergeant Sheridan, R.I.C., Food Inspector, Enniskillen, submitted to me a specimen of milk which contained 5.26 per cent. of non-fatty solids and 4.6 per cent. of fats. Assuming that the milk when pure contained the assumed minimal amount of non-fatty solids, 61 per cent. of its weight of water must have been added to it in order to have reduced the non-fatty solids to 5.26 per cent. I may here remark that it is the percentage of water added to milk, and not the percentage of added water in it, that should be stated in the certificate. In the above the certificate should state as follows: "This is milk to which at least 61 per cent. of its weight of water has been added as an adulterant, making thereby a mixture of 161 parts of milk and water."

A specimen which was examined a short time since for the Croom (co. Limerick) Creamery contained 5.2 per cent. of fats, but according to the 8.5 per cent. non-fatty solids standard it was adulterated with 19 per cent. of water.

DUBLIN, March 22, 1895.

The Use of Barium Thiosulphate in Standardizing Iodine Solution. R. T. Plimpton and J. C. Chorley. (*Proc. Chem. Soc.*, 149, p. 38).—For standardizing iodine solutions the authors recommend the use of barium thiosulphate, prepared by mixing warm solutions of 40 grammes barium chloride and 50 grammes sodium thiosulphate, each dissolved in 300 c.c. of water, the crystalline precipitate being washed and dried at 30°.

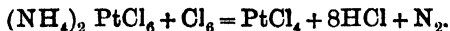
In the titration a weighed quantity of the $\text{BaS}_2\text{O}_3 \cdot \text{H}_2\text{O}$ is shaken with water in a stoppered bottle, and iodine run in until the salt is almost dissolved, when starch is added as a final indicator. Care must be taken to shake the bottle well after each addition of iodine.

The advantages claimed for the process are that barium thiosulphate, which has a high molecular weight, is easily obtained pure, keeps well, and reacts readily with iodine, while the progress of the reaction is marked by the gradual disappearance of the crystals.

C. A. M.

Uselessness of Kjeldahl's Method for Determining Nitrogen in Platino-Chlorides. Delephine. (*Bull. Soc. Chim.*, 1895 [8], xiii., 222).—A sample of trimethylamine platino-chloride gave 3.88 per cent. of nitrogen, instead of 5.15 per

cent., when treated by the Kjeldahl method as usually conducted, permanganate being used as an oxidant. Ammonium platino-chloride gave 1.89 per cent. of nitrogen instead of 6.26 per cent. The author attributes the deficiency of these results to the fact that platinum tetrachloride easily loses chlorine, which may be expected to decompose the ammonia or amine in the platino-chloride in some such manner as the following :



[The author does not appear to have tried to effect the determination without the use of a drastic oxidizing agent such as would certainly liberate chlorine in the reaction flask.]

A. G. B.

Colouring Matter in the Californian Red Wines. W. D. Bigelow. (*Jour. Amer. Chem. Soc.*, 1895, xvii., pp. 213-218.)—In addition to the difficulty of distinguishing some of the substances used for colouring wines from the natural colouring matter of the grape, there are the further difficulties that the colouring matter of wine changes with age, and that different reactions are obtained with wines of the same age, but of different varieties or from different districts. With the object of comparing the reactions obtained with Californian wines with those with European wines, the author examined ninety-four samples of the former, varying in age from one to seven years.

Three classes of reagents were used : 1. Those giving a red, blue, or violet colour with wines containing foreign colouring matter, and usually a green or grayish-green with pure wines ; for example, lead acetate, sodium carbonate, and bicarbonate borax and copper sulphate. 2. Certain metallic oxides (*e.g.*, manganese dioxide and lead dioxide) ; these were found to destroy almost all colour in Californian wines when used in the proportions recommended for French wines. 3. Methods in which chalk was used, treated with albumen and charged with various reagents.

The general conclusions arrived at were that in Californian wines the colouring matter was much more uniform than in European wines. The reactions were not always the same as with European wines, a gray or yellowish precipitate being sometimes obtained with reagents said to give green or grayish-green with French wines. On the other hand, no reactions were obtained which were characteristic of wines coloured with vegetable pigments.

C. A. M.

The Detection of Hydrogen Peroxide in Green Plants. A. Bach. (*Comptes Rendus*, 1894, cxix., 1218; through *Chem. Zeit.*)—The author, for the purpose named in the title, uses a reagent containing 0.03 grammes of potassium bichromate and 5 drops of aniline in 1 litre; 5 c.c. of this liquid are mixed with 5 c.c. of the solution to be tested, and to the mixture is added 1 drop of a 5 per cent. solution of oxalic acid. In the presence of hydrogen peroxide a red-violet coloration is produced. Tests made on 35 plants showed the presence of hydrogen peroxide in 18, while in 5 no reaction took place, and in the case of 2 the result was doubtful. The reaction is said to be recognisable in a solution containing 1 part of hydrogen peroxide in 1,400,000.

B. B.

Solvents for Perchromic Acid. W. M. Grosvenor, junr. (*Jour. Amer. Chem. Soc.*, 1894, xvii, 41; through *Chem. Zeit.*)—The author has examined the effect of a number of solvents on perchromic acid generated in the usual way from chromic acid and hydrogen peroxide. The following liquids do not dissolve perchromic acid: carbon disulphide, benzene, oil of turpentine, castor-oil, oil of winter-green, oil of bergamot, kerosene, paraffin-oil, chloroform, carbon tetrachloride, toluene, nitro-benzene and aniline. The following liquids, on the other hand, dissolve perchromic acid: ethyl acetate, ethyl valerianate, amyl valerianate, amyl chloride, amyl butyrate, amyl formate, amyl acetate. The colour is most stable in ethyl acetate, being permanent for 23 hours. The results are not entirely in accord with those of Griggi, who found that perchromic acid is more stable in amyl alcohol than in ether.

B. B.

The Detection of Arsenic in the Presence of Selenium. L. Dawydow. (*Farmazeft*, 1895, iii., 1; through *Chem. Zeit.*)—The so-called Bettendorf's reagent, that is to say an acid solution of stannous chloride, is useful for the detection of arsenic in quantities as small as 0.1 milligramme in 1 c.c. of water, but is not available in the presence of compounds containing such easily reducible substances as mercury, gold, tellurium and selenium, as these substances are separated in the elementary state. Thus a solution of selenious acid (1:1000) gives an immediate red precipitate with stannous chloride capable of masking a precipitate of arsenic; even with a solution ten times as dilute, a brownish coloration is produced. The presence of selenious acid retards the evolution of arseniuretted hydrogen in a Marsh's apparatus, and hinders the formation of an arsenical mirror. A series of experiments has shown that the definiteness of the mirrors obtained diminishes with increasing amount of selenious acid. For example, 1 milligramme of arsenious acid, when tested for in the presence of 18 milligrammes of selenious acid, failed to give a mirror even after one and a half hours, provided the evolution of hydrogen was slow; although when the stream of gas was rapid a mirror was obtained, the definiteness of which was favoured by leading a stream of hydrogen from an extraneous source through the Marsh's apparatus. When the proportion of arsenious acid was only $\frac{1}{100}$ of the selenious acid, no mirror could be obtained. The author has not proved whether this suppression of arseniuretted hydrogen is due to the formation of a compound of arsenic and selenious acid, or to the occurrence of the arsenic as solid arseniuretted hydrogen under the particular conditions obtaining. It is therefore advisable, in examining such materials as sulphuric acids and other sulphur compounds in which both arsenic and selenium may be present, to precipitate both by means of sulphuretted hydrogen, and to treat the resulting precipitate, consisting of sulphides of arsenic and selenium, together with free sulphur, with ammonium carbonate, thus removing the arsenic and allowing of its identification in the ordinary manner.

B. B.

Examination of Liquid Carbonic Acid. L. Grünhut. (*Chem. Zeit.*, 1895, xix., 505, 555.)—The examination of liquid carbonic acid is a matter which occasionally engages the attention of the commercial analyst, although but little has been pub-

lished on the subject. The only note dealing with the presence of impurities which has come to the notice of the author is one by Fleck, who stated that when strong, crude hydrochloric acid containing arsenic is used in preparing carbonic acid, some risk exists of arsenious chloride passing over into the compressors. The ordinary method of determining the freedom of the gas from gaseous impurities by absorption in caustic alkali solution, in the usual manner of gas analysis, needs no special description, and only requires the comment that in the author's experience the presence of more difficultly condensable gases hinders the liquefaction of the carbonic acid, and is therefore avoided by the manufacturer.

Other impurities have, however, been repeatedly detected. Liquid carbonic acid, although of apparently 100 per cent. purity, frequently has an objectionable smell, which impairs the quality of aerated liquids made by the aid of the gas. A product of this description gives a brownish colour to strong sulphuric acid, and slowly decolorizes dilute permanganate. The test with permanganate should be carried out both in acid and alkaline solution, as certain of the possible impurities in the carbonic acid react only under the one condition and certain others under the other. It is common to find a considerable amount of a brown turbid liquid left in the steel cylinders after the carbonic acid has been used. The author has obtained as much as 70 grammes of this impurity from cylinders originally holding 10 kilos of carbonic acid. On filtering this liquid a residue of from 1 to 2 per cent. of the weight of the liquid is separated, and consists of hydrated ferric oxide and a small quantity of organic matter. The filtered liquid deposits a considerable quantity of ferroso-ferric hydroxide on exposure to air, presumably from the oxidation of some iron compound which it holds in solution. When freed from this deposit, the final clear liquid is found to contain glycerin. The following analyses give some idea of the nature of the residual impurities found in carbonic acid cylinders, and indicate the variable character of this material :

	I.	II.	III.
Glycerin	0.81	0.33	6.63
Other organic matter	0.28	—	0.92
Ferrous oxide	0.45	0.40	0.84
Other mineral matter	0.03	0.07	0.24

The remainder of the material is presumably water, although no explicit statement to that effect is made. The origin of the glycerin is not obvious, but it is possibly used in the compressors, and carried over into the carbonic acid receiver. Much more important as regards the flavour of the product are the iron compounds, the presence of which cannot be entirely accounted for by the action of the water saturated with carbonic acid which finds its way into the cylinders, and is free to act upon their walls. By direct experiment the maximum quantity of iron that can be held in solution as ferrous carbonate by water charged with carbonic acid has been found to be equivalent to 0.056 per cent. of FeO. Contrasted with this is the fact that the residues from the three cylinders examined contained 7 to 15 times as much iron as is possible on the assumption that the metal is present as ferrous carbonate. It is probable that glycerin itself has a solvent action, not by direct attack of the iron, but by increasing the solubility of ferrous carbonate formed in the ordinary manner.

The author considers that the detection of this dirty ferruginous liquid containing glycerin in the cylinders which he has examined, affords a basis for explaining the cause of the disagreeable taste and smell of the carbonic acid and of aerated liquids prepared from it. The gas as it escapes from the cylinder must be charged with a fine spray of the impure liquid, the proportion of impurity increasing as the contents of the cylinder diminishes, owing to the fact that the impure liquid is heavier than liquid CO_2 , and therefore remains at the bottom of the cylinder comparatively undisturbed until the bulk of the gas has been drawn off. The observations here recorded point strongly to the necessity for more careful scrubbing and drying of the carbonic acid before it is bottled, and make it incumbent on the technical chemist not to content himself with a determination of the substantial purity of the carbonic acid itself, but also to make a special determination of any residual liquid that may remain in the cylinder after the gas has been blown off.

B. B.

Simplified Method for Determining Phosphoric Acid by Means of Molybdate Solution. J. Hanamann. (*Chem. Zeit.*, 1895., xix., 553, 554.)—Where speed is not a prime necessity, separation of phosphoric acid by means of molybdate solution and subsequent conversion into magnesium pyrophosphate must remain the standard method. For rapid determinations, however, any process which permits of obtaining the yellow precipitate of phosphomolybdate of constant composition is worthy of attention. The main difficulty in the way of such a process consists in the tendency of ordinary molybdate solution to deposit molybdic acid on heating. The difficulty can be met either by using a molybdate solution, from which all superfluous molybdic acid has been separated by repeated boiling, or by adopting a molybdic solution of such a character that it can be relied upon to precipitate phosphoric acid in the cold, the precipitation being, of course, aided by stirring. A molybdic solution possessing this quality is one containing 100 grammes of molybdic acid, 1 litre of 10 per cent. ammonia and 1.5 litre of nitric acid having a specific gravity of 1.246. Solutions containing phosphoric acid are completely precipitated by this molybdic mixture by half an hour's vigorous stirring in the cold. The precipitate can be ignited until it acquires a pure blue-black colour, when it will contain 4.018 per cent. of phosphoric acid. In carrying out this process the bulk of the precipitate should be removed from the filter, which should be ignited separately. If a first ignition fail to give a mass of uniform blue-black colour, the precipitate should be moistened with ammonia, dried, and ignited again. The ignition preferably should be conducted in platinum, with the crucible resting on a piece of platinum gauze, which should glow red-hot while the bottom of the crucible is not visibly red.

B. B.

The Use of the Mohr-Westphal Balance in Milk Analysis. C. H. Wolff. (*Zeit. für angewand. Chemie*, 1895, Heft v., pp. 134-137.)—By a modification of the Liebermann-Székely method the Mohr-Westphal balance can be used for the direct estimation of the percentage of fat in milk, as well as for the specific gravity. Fifty grammes of the milk are well shaken in a well-corked 200 c.c. flask with 5 c.c.

of KOH of 1.27 specific gravity. Fifty c.c. of petroleum ether (specific gravity 0.663) are then added and the mixture well shaken till an emulsion is formed, when 50 c.c. of alcohol (95 to 96 per cent.) are added and the shaking repeated four or five times, the flask being allowed to stand four or five minutes between each shaking. Twenty c.c. of the clear petroleum-ether layer, which now contains all the fat, are removed by a pipette, with its upper tube bent at an obtuse angle, and its lower tube passing through a cork, which fits the 200 c.c. flask. The 20 c.c. are evaporated in a previously weighed flask on a sand-bath or hot plate, and the residual fat determined directly in percentage on the Westphal balance. For weighing a small stirrup scale and three riders (2.0 gramme, 0.20 gramme, and 0.02 gramme) are required. The empty fat flask and scale are substituted for the sinking weight of the balance, and should be so counterpoised that the addition of the 2 gramme rider below the flask produces equilibrium. When weighing the residue of fat, if the removal of this 2 gramme rider produce equilibrium, the amount of fat must be 2 grammes, which would correspond to 10 per cent. But since the percentage of fat is always less than 10, it is necessary, after removal of the 2 gramme rider, to use the three riders on the beam in the usual way, and to deduct the sum of the numbers obtained from 10 per cent. Thus the amount of fat in the flask from 20 c.c. of petroleum ether (= 20 c.c. of milk) was 0.855 gramme, which, multiplied by five, gave 4.275 per cent. On the Westphal balance the riders gave the figures 5.725, which, deducted from 10 per cent., left 4.275 per cent. The results tabulated show that the method agrees with the ordinary analytical methods to the third place of decimals.

C. A. M.

Separation of Nickel and Iron. E. D. Campbell and W. H. Andrews. (*Amer. Chem. Journ.*, 1895, xvii., pp. 164-167.)—In many processes used to separate iron and nickel, the iron is precipitated first, and carries down with it more or less nickel. To avoid dealing with a bulky iron precipitate, several methods have been proposed in which nickel is precipitated first.

Classen* precipitates nickel as double potassium nickel oxalate in acetic solution, the double oxalate of iron being kept in solution.

Moore† converts both into double cyanides with KCN, and precipitates the nickel with KOH and Br.

Leroy‡ and Vortmann§ recommend electrolytic methods.

The authors have found the following method of separation accurate and easy. The mixed metals are dissolved in HNO₃ in an Erlenmeyer flask, an excess of 20 to 25 c.c. of the acid being used, and to this is added a solution of sodium pyrophosphate in warm water in the proportion of about 13 grammes of pyrophosphate for each gramme of the metal. The white ferric pyrophosphate which precipitates is brought into solution by adding cautiously a moderately concentrated solution of Na₂CO₃ until it just dissolves. The solution is filtered through asbestos into a 500 c.c. flask, and the nickel precipitated as brick-red nickelous xanthate by adding to the cold

* *Zeit. Anal. Chem.*, 1879, pp. 189-193.

† *Chem. News*, 1891, vol. lxi., p. 194.

‡ *Chem. News*, 1887, vol. lvi., p. 3.

§ *Monat.*, 1898, vol. xiv., p. 587.

solution 2 grammes of potassium xanthate freshly dissolved in a little water, the stoppered flask being shaken for ten minutes to complete the precipitation. The precipitate is then filtered off on asbestos, washed immediately with a 1 per cent. solution of potassium xanthate, and dissolved off the filter with freshly diluted (1 : 1) fuming HNO_3 .

Two c.c. of slightly diluted H_2SO_4 are added to the filtrate, and the liquid boiled till HNO_3 is completely expelled and fumes of sulphuric anhydride begin to appear. The nickel sulphate thus obtained is dissolved in a small quantity of water, and the iron present precipitated by a slight excess of ammonia. The ferric hydroxide is filtered off, dissolved in H_2SO_4 and reprecipitated with ammonia, the filtrate being added to the former filtrate. Even when much iron was originally present, the ferric hydroxide from the nickel solution rarely exceeds 0.003 gramme. The nickel in the solution of nickel sulphate may then be determined, either electrolytically by transferring it to a large platinum dish, adding 3 grammes of disodic-hydric phosphate together with 25 c.c. of strong NH_4OH , and depositing the metal by means of a current of about 0.14 amperes per 100 sq. cm. of the dish for twelve hours, or a volumetric method may be used. In the latter the nickel is determined by means of a standard solution of KCN, with AgNO_3 , followed by KI as an indicator, the suspended silver iodide giving an opalescence to the solution, which does not clear up until all the nickel has been converted into potassium nickel double cyanide.

The following table shows the accuracy of the method :

Iron added. Grammes.	Nickel added. Grammes.	Nickel recovered. Grammes.	Gain or loss. Grammes.	Method.
0.2105	0.08892	0.08910	+ 0.00018	E
1.0000	0.03430	0.03435	+ 0.00005	
1.0041	0.05298	0.05345	+ 0.00047	
1.0043	0.10824	0.10825	+ 0.00001	Vola.
2.0043	0.02690	0.02740	+ 0.00050	
1.0010	0.00100	0.00089	- 0.00011	Volum.
1.0086	0.06578	0.06532	- 0.00046	
1.0066	0.07582	0.07583	+ 0.00001	
1.0057	0.08017	0.08050	+ 0.00033	
1.0071	0.09389	0.09381	- 0.00008	

C. A. M.

Extracts from the Proposed Austrian Alimentary Code. Wine. (*Bull. de l'Assoc. belge des Chimistes*, 1895, pp. 177-182.)—The substances which are allowed to be added before or after fermentation are: (a) Grapes fresh and dried, or their juice. (b) Alcohol pure, and some cognac. In grape wines the quantity of added alcohol must not exceed 2 per cent., and the total quantity of alcohol must not be more than 16 per cent. by volume. The following wines are exceptions to this: Old Tokay, 18 per cent. (Sweet Tokay, 16 per cent.); Malaga, 18 per cent.; Greek, Cyprian, Asiatic, Californian, and Cape wines, 20 per cent.; Australian, 21 per cent.; Port, 23 per cent.; sherry and Marsala, 25 per cent. In Sicilian wines the maximum of 27 per cent. is quite exceptional. (c) Cane-sugar and invert sugar. (d) Pure carbonic

acid. (e) Pure calcium carbonate. (f) Clarifiers not injurious to health, such as tannin, egg albumen, etc. Substances prohibited are: Alumina and magnesia preparations, sulphites, mineral acids, colouring matters from tar and other foreign materials, glucose, molasses, cane and invert sugar (impure), impure alcohol, salicylic acid, glycerin, aromatic bodies and substances injurious to health.

In judging as to the purity of a wine of a special vintage, it is necessary to base one's conclusion on the analysis of wines from the same district and of the same age.

Extract.—Wines completely fermented should yield at least 14 grammes per litre. Those containing less than that amount are to be suspected unless it be found normal for wines of the same kind and age. After removing the fixed acids, the residue should still be 11 grammes per litre.

Mineral Matter.—At least 1·3 grammes per litre in normal wines.

Ash.—Should not, as a rule, exceed $\frac{1}{10}$ of the total extract.

Glycerin.—The relation to the alcohol is from 7 to 14 per cent.

Free Acid.—In natural wines, containing about 8 grammes of acid per litre, the proportion of free tartaric to the fixed acids is as 1 to 5 or 6.

Sodium Chloride.—Not more than 0·05 grammes per 100 c.c., unless the wine come from a district with soils rich in chlorides.

Sulphuric Acid.—0·92 gramme of SO_3 per litre is the usual maximum.

Phosphoric Acid.—In certain cases gives useful indications, e.g., in the case of medicinal wines (Austrian and Hungarian *vins de santé* contain at least 0·6 gramme P_2O_5).

Nitrogen.—According to researches made at Klosterneuberg experimental station, natural wines rarely contain less than 0·07 gramme, or more than 0·08 gramme per litre, though in certain isolated cases it has been found as high as 1·35 gramme. Less than 0·07 gramme is suspicious.

Nitric Acid.—Only suspicious when the diphenylamine reaction is very marked.

Sulphurous Acid.—According to the medical faculty of the University of Vienna, a wine containing more than 0·008 gramme per litre is to be condemned.

Most of the preceding estimations are made by the usual methods, and the only points calling for special notice are the determination of the extract and the search for foreign colouring matters. In wines containing up to 3 per cent. of extract, 50 c.c. are evaporated on the water-bath in a platinum basin and dried for two and a half hours. Those richer in extract are diluted until they do not contain more than 3 per cent. and treated in the same way. In the case of sweet wines it is preferable to obtain the extract from the specific weight, for which purpose Balling's table is serviceable.

In examining for vegetable colouring matters the wine is treated with an excess of lead acetate. The colour of the precipitate obtained with natural wines is bluish-gray to bluish-green. With the colouring matter of the bilberry a mauve colour is obtained, and with that of the elder a greenish tint. With the colouring matter from the leaves of the cochineal oak (*Quercus coccifera*) the colour is reddish-violet.

In searching for coal-tar colours the lead acetate precipitate is filtered, the filtrate agitated with amyl alcohol, and the coloured amyl alcohol examined; 100 c.c. of the wine should be used, and 30 c.c. of lead acetate.

C. A. M.

REVIEW.

CHEMICAL ANALYSIS OF OILS, FATS, AND WAXES, AND OF THE COMMERCIAL PRODUCTS DERIVED THEREFROM. From the German of Professor Dr. R. BENEDIKT. Revised and enlarged by J. LEWKOWITSCH. (Macmillan and Co., 1895. £1 1s.)

Before entering into a discussion of the contents of this valuable work, we must take exception to the English editor's opening sentence in the preface, that "there has not hitherto been any English work dealing especially with the chemical analysis of oils, fats, and waxes." One of the volumes of Mr. Allen's great work on "Commercial Organic Analysis" deals most "especially" with the subject. Dr. Alder Wright's work, which, by the way, has the right of priority to the title chosen by Dr. Lewkowitsch, and which was published only last year, also deals "especially" with it, to say nothing of the numerous fairly complete monographs inserted in several chemical encyclopedias and dictionaries. The English editor of the work under review might have given credit where credit was due. To Mr. Allen in particular we owe so much of our knowledge of the composition, and what is the same, of the analysis of oils, that no writer for years to come can afford even to appear to overlook his work.

The reader cannot fail to be struck with the fact that of late years by far the greater part of the advance which has been made in the knowledge of the chemistry of oils is due to the analytical chemist, and not to the academical investigator. With the exception of the work of Hazura and his coadjutors (a work which stands in urgent need of confirmation and development), no very important addition has been made to the broad theoretical foundations laid long since by Chevreul and Liebig and their pupils, while, on the other hand, the analyst has most industriously multiplied and developed his methods. The measure of the proportions of free fatty acids, and of the amount of alkali requisite for neutralizing the total fatty acids, the measure of the proportions of the glycerides, volatile fatty acids, the insoluble fatty acids, hydroxyacids, and of the unsaturated fatty acids, with the isolation and determination of the alcohols, and other unsaponifiable matters, are all advances made during the last twenty years, and most of them are of still more recent origin. These now supply analytical data of infinitely higher scientific value than the old colour reactions and physical differences with which the last generation of chemists contented themselves.

The work deals most fully and clearly with the determination of these "constants" of fatty and allied bodies, and with their interpretation.

From the general part of the work, the intelligent student will at once realize that there is still need for a vast amount of further investigation. Essentials, like the separation of unsaturated from saturated acids, the determination of one saturated fatty acid in the presence of others, the quantitative estimation of the various groups of the unsaturated fatty acids, are even now almost in embryo, and until these, and other allied problems are fully solved, accurate fat analysis will remain an impossibility. Indirect methods, it is true, exist in plenty, but none of these are beyond criticism, and many involve assumptions which are unwarranted. Thus

the indirect determination of palmitic and stearic acids from the molecular weight, as deduced from the titration of a mixture, presupposes that only the two acids named are present, and the proof of this is hardly ever obtainable. More complex still is the problem when unsaturated acids are present. The iodine number is often translated into percentages of oleic acid; but this is obviously wrong, unless there are no acids with less hydrogen than oleic present, yet no adequate means are at hand to prove their absence.

The special parts of the work, dealing with the constants and properties of individual oils, fats, and waxes, are very complete, and contain an immense amount of information. The tabular statements interspersed throughout this part are particularly valuable, and in this respect the work is an improvement on the original edition of Benedikt. Never before has so large a mass of similar material, for the most part trustworthy, been collected together. Had the authors done no other work than thus to collect data, they would have laid chemists under a great obligation; but when the whole scope of the work is considered, it can safely be said that no chemist who is called on to analyse fatty bodies can afford to be without the book. We must specially mention its value to the public analyst, though we are sorry to see that *THE ANALYST* is a journal apparently little known either to the author or his translator. Several important contributions, especially on the subjects of butter and lard, which were originally published in *THE ANALYST*, are attributed to other journals, erroneously described, or wholly ignored. As regards butter, the reference to adulteration with clay, chalk, gypsum, and flour, might with advantage have been omitted. The conversion of Reichert numbers into Reichert-Wollny figures by multiplication of the former by 2 is unjustifiable, the factor being more nearly 2.2.

There are a few errors in the book, taken over by the English editor from the German author. Thus, in Herbig's modification of the Benedikt-Zigmondy method for the determination of glycerin (p. 163), the amount of permanganate requisite is not clearly given—6.87 parts for one of glycerin are insufficient, half as much again being absolutely necessary, or the results will be found too low in almost all cases.

The method for the analysis of beeswax, depending upon the determination of the acid value and the ether value, is erroneously attributed to Hübl; and it is further stated that "Hehner adopts Hübl's method, with the only difference that he substitutes methyl alcohol for ethyl alcohol," etc. Hübl's paper on wax analysis was published in the September number of Dingler's *Polytechnisches Journal*, 1883, the date of the writing of the paper being July, 1883. Hehner's paper was read before the January meeting of the Society of Public Analysts, and was published in the February number of *THE ANALYST*, 1883, six months before the appearance of Hübl's paper. It is quite possible that Hübl's work on the subject was quite independent, but it was certainly not antecedent. Benedikt, like most other German authors, not understanding the mysteries of methylated spirit—a product peculiar to England, and a curse to English chemists—translates methylated spirit as "methyl alcohol." But the English editor might have known better than to repeat the error, which a glance at the original paper would have prevented.

But these mistakes and omissions are trifles compared with the mass of sound information contained in the book, which we cordially recommend to the readers of *THE ANALYST*, partly on account of its intrinsic value, but more strongly still on account of the help and stimulus it must prove to investigators of a particularly difficult and important division of chemistry.

O. H.

THE ANALYST.

JUNE, 1895.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday evening, May 1. The chair was taken by the President (Dr. Thomas Stevenson).

The minutes of the previous meeting were read and confirmed.

The following gentlemen were elected members of the Society: Mr. H. Irving Foster, The Elms, Anlaby Road, Hull, and Mr. Alec Poole Wilson, Knocklong, Co. Limerick.

Mr. Wynter Blyth then read the following paper:

THE IDENTIFICATION AND ESTIMATION OF CARBOHYDRATES IN MILK.

By A. WYNTER BLYTH.

THE importance of determining in every milk analysis the milk-sugar may be at once conceded, when it is remembered that it constitutes almost a third of the weight of the total solids, that it is the first substance that suffers change by decomposition, and that it is mainly the cause of discrepancies in analyses of stale milks. Experience has also shown that insoluble adulterants are but rarely added to milks; the greatest number are to be looked for in the fluid portion freed from albuminoids and fat. The old method of dissolving out the more soluble portions of milk residues by treating the dried total solids first by ether, and then by dilute alcohol, is both tedious and of small accuracy.

The best method is, after many trials, I am convinced, a physical one, supplemented by a copper process; and lastly, when necessary, obtaining the osazone.

The first step is to prepare a clear whey. An excellent paper on this subject was published so far back as 1887 by Dr. W. Wiley, the chief Chemist for the U.S. Department of Agriculture.* He used the following reagents for the precipitation of the albuminoids:

- (1) Saturated solution of basic lead acetate.
- (2) Nitric acid solution of mercuric nitrate diluted with an equal volume of water.
- (3) Acetic acid, specific gravity 1.040.
- (4) Nitric acid, specific gravity 1.197.
- (5) Sulphuric acid, specific gravity 1.255.
- (6) Saturated solution of sodium chloride.
- (7) Saturated solution of magnesium sulphate.

* Foods and Food Adulteration, United States Department of Agriculture, Bulletin No. 13.

(8) Solution of mercuric iodide in acetic acid, a litre of the solution being made with 161 grammes HgCl_2 , 395 grammes KI, and 228 c.c. of acetic acid.

Dr. Wiley also used a number of other precipitating agencies, but finally gave the preference to two solutions, viz., to the mercuric nitrate, or to the mercuric iodide solution.

These solutions he used as follows: he added 1 c.c. of the mercuric nitrate, or 30 c.c. of the mercuric iodide solution, to certain quantities of milk, varying these quantities according to specific gravity. For a milk of specific gravity 1.026 he took 60.5 c.c. of the milk; if of specific gravity 1.030, 60 c.c. of milk; and if of specific gravity 1.034, 59.5 c.c. of milk. The precipitated albuminoids he considered to occupy a bulk of 2.4 c.c., so that the volume in each case was made up to 102.4 c.c.

The milk thus prepared was shaken, filtered, and the filtrate polarized.

The instrument used by Wiley was a Laurent large model polarimeter. The specific rotatory power of milk-sugar for the sodium light he considered to be 53° .

I have tried both the mercuric nitrate and the mercuric iodide precipitants, and both give good results; but the presence of mercury in the filtrates is objectionable, for the filtrate can only be used for the single purpose of polarimetrical observation. For shadow instruments you can get quite as clear and bright a filtrate from the use of copper sulphate, and if only the proper quantity is taken the filtrate is of so pale a blue that it in no way interferes with the reading; besides which, such a liquid can be treated with Fehling, and the amount of copper suboxide reduced ascertained by weighing.

I have, however, finally adopted a method of precipitating by acetic acid as follows: 25 c.c. of milk are diluted to about 50 c.c. with distilled water, and then strong acetic acid is added drop by drop until the casein begins to separate. The liquid is then heated to boiling, and while still hot, "whirled" in a glass cylinder by the aid of a centrifugal machine. The casein, coagulated serum, albumin, and fat separate and collect in a more or less firm coagulum at the bottom of the cylinder, and the supernatant fluid is easily filtered; the precipitate is also transferred to the filter and washed with hot water. Finally the filtrate is cooled, made up to 100 c.c., and submitted to the processes to be detailed.

The filtrate is usually in a normal milk of a feeble yellow colour, but perfectly bright, and suitable for testing for substances such as salicylic acid, borax, dextrin, or any soluble addition. It is also most suitable for optical methods, and for any estimation of sugar by the reduction of cupric oxide. It will be obvious that such a solution should contain somewhere near 1 per cent. of hydrous milk sugar.

The saccharometer I use is a Laurent half-shadow arranged for ordinary white light, and the source of illumination has been a "Welsbach" incandescent gas-flame. The saccharometer has a straight index with vernier, and 100° on the scale is equal to a 16.2 per cent. of sugar when viewed in the 20 centimetre tube. The optical power of sugars generally is not the same for strong as for dilute solutions, so that as a 1 per cent. solution is in all these cases taken, it is essential that the instrument be standardized with the same solution as is used for standardizing the copper process. For example, in my own instrument, 10.28 per cent. milk-sugar in a 400 m.m. tube marks 100° on the scale, but a 1 per cent. marks 10° ; similarly, 1 per

cent. of cane-sugar, or 1 per cent. of dextrose, all have for such dilute solutions a slightly different value at ordinary temperatures—from 15° up to 18° —than stronger solutions.

If cane-sugar is large in amount relatively, the optical supplemented by the copper process at once reveals it, for we have in this case two sugars, both having an action on polarized light in different proportions, and the one having an action on copper, the other but little action on copper. Thus, a milk which was prepared so as to have practically equal parts of cane and milk sugar, containing in 100 c.c. 3.48 of cane-sugar and 3.52 of milk-sugar, gave by the saccharometer an indication of 8 per cent. of milk-sugar; by copper an indication of 3.9 per cent. of milk-sugar, so that, without inversion, it would be evident that in this case a sugar was present that rotated strongly, and only acted on copper slightly; and dextrin having been proved absent by testing the clear liquid with iodine, there would not be much error in diagnosing the presence of cane-sugar.

With smaller proportions of cane to milk, the differences are not so great, and inversion is necessary. Inversion of the whey of pure milks, calculated, not into galactose, but lactose, makes an apparent difference of from 0.5 to 0.8 per cent.; thus, a milk which by the optical method showed 4.11 per cent. of milk-sugar, and by copper 4.23 per cent., on inversion (by HCl) showed 3.5 per cent. milk-sugar at 16° .

This change in the optical power of milk-sugar on inversion with HCl renders the ordinary method of calculating the proportion of cane-sugar in these dilute solutions inapplicable.

Nevertheless, as a means of diagnosis inversion is valuable. A milk was prepared containing in 100 c.c. 3.73 per cent. of milk-sugar and 0.89 per cent. of cane-sugar.

In the 400 m.m. tube of my instrument, this should rotate, when diluted as in the process given, as follows:

0.93 of milk-sugar	9.3 degrees
0.27 of cane-sugar should mark	2.00 degrees

11.3

The actual degrees found were, however, a little higher, viz., 11.7, and this would be considered to be equal to 4.68, or, say, 4.7, of milk-sugar; copper indicated 5.0 per cent. of milk-sugar; on inversion, the copper value rose to 6.6 per cent. of milk-sugar, and the polarimetric reading at 18.0 was 6.3 degrees, showing a reduction of 5.42 degrees.

If glucose be added to milk, we then have the problem of two sugars, which act unequally to polarized light and which act unequally towards copper, thus, with the same proportions as in the last case, that is to say, or nearly so, on a milk containing 0.9 glucose and 3.73 milk-sugar, the saccharometer indicated 4.4 milk-sugar, and the copper 6.2, a difference between the optical and the copper processes of 1.8 per cent.

The Determination of Sugar by Copper.—In my earlier experiments, this was done by weight, a known quantity of the filtrate being boiled up with an excess of Fehling, the hot liquid transferred to a cylinder, well "whirled," the supernatant fluid being decanted, the copper suboxide washed by decantation with hot water until the water was no longer alkaline, and then the cylinder with the copper oxide dried and weighed.

In this way the difficulty of filtration was avoided, and the method was fairly speedy, and good results were obtained; the difference between the two determinations expressed as milk-sugar varying from 0.1 to 0.19 per cent.

Since, however, the publication by Mr. Gerrard* of his improved cupric cyanide process, the determination by weight has been abandoned, and the milk-sugar is estimated as follows: To 10 c.c. of a copper solution (Fehling's strength) 10 c.c. of the alkaline tartrate are added, and the liquid still further diluted with 40 c.c. of water, heated to boiling, and then drop by drop a 5 per cent. solution of potassic cyanide added, until there is only the faintest blue colour to be observed. A second quantity of copper and alkaline tartrate is then added, and, while boiling, the same 1 per cent. solution of milk-sugar, diluted one half, which has been used for standardizing the saccharometer, run in slowly from a burette to complete decolorization. Having made two or more experiments with the pure solution of known strength, it is obvious the value of the Fehling in terms of milk-sugar is accurately known; 50 c.c. of the already diluted milk-whey are made up to 100 c.c., and this is run into the boiling Fehling as before until the blue liquid is decolorized. The end reaction is quite definite and sharp. Soxhlet has laid it down that, as regards the ordinary determination by Fehling of milk-sugar, complete reduction does not take place under some six minutes' boiling. This observation does not apply to the cupric cyanide process; the decolorization attains its maximum for a given quantity of milk-sugar solution of the strength named well within a minute and a half.

The following table gives an example of a few milks which have been "whirled," the fat determined by the centrifugal machine, the specific gravity taken by a hydrometer, the total solids calculated by the Richmond scale, and the milk-sugar determined both by copper and by the polarimeter:

ANALYSES OF COMMERCIAL MILKS.

Specific Gravity.		Total Solids.		Fat.		Polarimeter.		Milk-sugar.	
								Cyanide	Copper Process
1031	...	10.4	...	2.2	...	4.08	...	4.31	
1034	...	12.68	...	3.5	...	5.00	...	4.90	
1032	...	11.1	...	3.0	...	4.24	...	4.31	
1033	...	12.9	...	3.8	...	4.24	...	4.26	
1032	...	12.3	...	3.6	...	4.04	...	4.56	
1034	...	12.55	...	3.4	...	4.40	...	4.24	
1033	...	11.85	...	3.0	...	4.24	...	4.23	
1033	...	12.78	...	3.8	...	4.72	...	4.92	
1031	...	11.8	...	3.4	...	4.20	...	4.26	

Hence, it is abundantly clear that, should there be an appreciable difference between the presumed percentage of milk-sugar, as determined by optical and copper processes, the carbo-hydrates of the milk are abnormal, and there is a probability of either dextrin or cane-sugar, or glucose, having been added, and it is necessary to examine further.

Dextrin may be dismissed in a sentence, for the clear filtrate, when tested with a

* *Pharm. Journal*, 3rd Series, vol. xxv., 912.

droplet of iodine, at once gives the well-known reddish colour, so that its qualitative detection is as simple as possible.

To identify with certainty foreign sugars, it is well to obtain the osazone. Twenty-five c.c. of the milk are diluted, as previously described, and a filtrate obtained, free from serum albumin casein and fat, neutralized and concentrated down to 30 c.c. During this process an albuminoid settles and adheres to the bottom of the dish, which is separated by filtering through a very small filter; to the clear filtrate 2 grammes of sodic acetate and 1.5 grammes of phenylhydrazin hydrochlorate are added, and the whole heated in a flask in a water-bath for one hour and a half. On cooling, the liquid becomes almost solid: the crystalline magma is collected on a filter, washed once with a little cold distilled water; the crystals are pressed between blotting-paper and dried in a water-oven. The osazone is then boiled with absolute alcohol, just sufficient to dissolve it; this, for the quantity taken, is usually about 30 c.c. to 40 c.c.; the alcoholic liquid is allowed to stand over night to see if any crystals separate. Should glucose or invert sugar have been present, well-defined crystals will have separated; if milk-sugar alone was present, there may be a slight amorphous deposit at the bottom of the flask, but no crystals visible to the unassisted sight.

The purification of the lactosazone is now proceeded with. The alcoholic solution is placed on the water-bath and heated to boiling, and while boiling hot water is added little by little until a slight turbidity shows itself; a good portion of the alcohol is now allowed to evaporate slowly away, and on placing the liquid in a cool place the osazone crystallizes out. Lactosazone derived from such a precipitation is coloured, and, microscopically examined, is seen to consist of aggregations of warty masses, some of which are strikingly like starch granules, exhibiting a split hilum with radiating fissures; this appearance is constant and characteristic, but it is also a sign that the osazone is not pure. Such crystals melt at various temperatures, according to purity, from 190° to 195° . To obtain it pure, the process of dissolving in alcohol, precipitating by hot water, pressing the crystals between blotting-paper, drying, etc., will have to be done at least three times. When pure the crystalline appearance is very similar, but not identical to that of glucosazone, viz., needle-like crystals—these melt at 200° or a little above. Since one of the main methods of distinguishing between the osazones is their solubility or insolubility, I have made a few observations on the solubility of lactosazone.

If lactosazone is shaken up with water and allowed to stand for twenty-four hours, then 100 c.c. of water dissolves 20 milligrammes. If 14 c.c. of a saturated solution in alcohol be poured into 100 c.c. of water and allowed to deposit, every 100 c.c. of the filtered solution hold in solution 34.8 milligrammes. Lactosazone is freely soluble in hot alcohol, and none separates on cooling, unless the solution be highly concentrated.

On the other hand, glucosazone requires repeated boiling in considerable quantities of absolute alcohol before it dissolves, and on cooling most of the osazone separates out 100 c.c. of 98 per cent. alcohol, only retaining, at ordinary temperatures, 30 milligrammes.* The separation is in fine crystals. These crystals, without further purification, melt at or about 197° ; while, as before remarked, the impure osazone from

* 100 c.c. of cold water only retain 11 milligrammes of glucosazone in solution.

milk-sugar melts from 190° to 195° ; glucosazone can therefore be rapidly purified by two or three crystallizations from alcohol; it then has a melting-point of 203° to 204° . Glucosazone is always in the form of needles, most of which are in bundles. No warty or starch-like appearance has been observed. A mixture of glucosazone and lactosazone may be separated in great part by fractional crystallization.

Cane-sugar gives no osazone until inverted; it then gives an osazone not to be distinguished from glucosazone; so that the diagnosis of a milk adulterated with cane-sugar is:

(1) The considerable want of agreement between the results from the copper process and the polarimeter readings.

(2) Lactosazone is alone obtained by treatment with phenylhydrazin.

(3) On inversion the considerable rise in the amount of copper reduced.

ADDENDUM.—Since I had the advantage of hearing the discussion on the foregoing paper, I have had made by my assistant, Mr. W. A. Rogers, a few experiments on the citric acid method of inversion. Fifty c.c. of a 1 per cent. solution of milk-sugar gave an optical value in the 400 m.m. tube of 10.1° , and 6.3 c.c. decolorized the hot cupric cyanide. On heating to boiling for nearly twenty minutes the same solution with half a gramme of citric acid, replacing the evaporated water until it assumed its original bulk at 15° ; the optical value was unchanged, and the c.c. used to decolorize the copper were 6.6, so that I conclude that no change took place in the milk-sugar. On the other hand, 100 c.c. of a 1 per cent. solution of milk-sugar acidified with 2 grammes of citric acid, and concentrated down to 30 c.c. on the water-bath, suffered change, and a portion was converted into galactose; a small amount of a crystalline osazone was separated, which, after several crystallizations, melted at 197.5° , and was probably not quite pure galactosazone.

A hundred c.c. of 1 per cent. solution of cane-sugar was rapidly inverted by the addition of 1 gramme of citric acid and heating for 20 minutes.

Fifty c.c. of a 1 per cent. solution of milk-sugar and 50 c.c. of a 2 per cent. solution of cane-sugar mixed, gave, before inversion, an optical value of $+16.25^{\circ}$; after inversion with citric acid an optical value at 18° of $+3.6^{\circ}$. Before inversion the number of c.c. consumed of the solution to decolorize the copper was 14.2; the number after, 3.8 c.c. Hence without a doubt the citric acid method promises well, and if the acid is not added in excess and the heating not prolonged, the milk-sugar is not affected.

DISCUSSION.

The President having invited discussion,

Mr. RICHMOND said that his experience had led him to prefer Dr. Vieth's modification of Dr. Wiley's method of preparing the whey, as being more accurate. One great advantage of this method was, that the fact of the mercuric nitrate being added in the proportion of 3 c.c. to 100 c.c. of the milk caused the whey to occupy the same volume as the original milk less that of the fat, since, for all practical purposes, 3 c.c. might be taken as the volume of the albuminoids precipitated.

With regard to polarimeters, he had found that the instrument described and used by Dr. Vieth gave sufficiently good results, and its price was very much lower than that of the instrument mentioned by Mr. Blyth. As the former instrument was

in the possession of a good many analysts, he would like to point out a small error occurring in Dr. Vieth's description of it. It was stated that the instrument might be taken as reading the percentages of milk-sugar direct; this, however, was only true speaking broadly; to be more accurate, the reading should be divided by 1.042. If, however, Mr. Blyth's plan of standardizing from a solution of known strength were adopted, this discrepancy would not be found.

He thought it might be useful, in estimating sugar other than milk-sugar, to bear in mind the ratio—pointed out many years ago by Dr. Vieth—between the ash, albuminoids and milk-sugar, viz., 2 : 9 : 13, which had been found to hold good to an extent that might almost be called remarkable. For the estimation of cane-sugar there was Kjeldahl's process of inversion by means of invertase, which only affected the cane-sugar, and not the milk-sugar, so that if a solution were made and treated with invertase, and any change in the rotating powers was observed, it might be regarded as certain that cane-sugar was present.

Mr. Blyth relied on the separation of the osazones for the detection of glucoses, but the anhydride of phenyl-lactosazone (lactose being the only sugar which gave an anhydride) would probably interfere with this reaction.

Mr. ALLEN said that, many years ago, before the value of the solids-not-fat was recognised, it was his custom to determine the specific gravity of the whey of milk. For this purpose about 100 cc. of the sample were heated to about 120° C., and then five or six drops of acetic acid added without agitation. On keeping the liquid at rest for eight or ten minutes the curd separated perfectly, so that with care nearly the whole of the whey could be syphoned off. If once the liquid was agitated, it was found impossible to obtain a clear filtrate.

He (Mr. Allen) had made a number of experiments with Gerrard's cyano-cupric process of titrating glucose. In its original form the method was quite unworkable, but the improved process, described in the *Pharmaceutical Journal* for April 20, which he might say was practically worked out jointly by Mr. Gerrard and himself, gave excellent results. He regarded this as the best of the copper methods of titrating glucose, but had had no experience of it in the titration of milk-sugar, and should like to be quite sure that the same ratio of reduction applied as in the ordinary Fehling's or Pavy's test before he accepted the indications of the process as correct.

In working with the osazones, he had observed that glucosazone could be readily extracted from acidulated aqueous liquids by agitation with ether, which fact might be used for its purification.

Mr. Wynter Blyth stated in his paper that he detected cane-sugar in presence of milk-sugar by boiling the mixture with dilute acid, which converted the former sugar into a mixture of dextrose and lævulose, and the latter into galactose. He then separated these by taking advantage of the different solubilities of their osazones in alcohol. But this process entirely ignored the fact that dextrose was a product, together with galactose, of the inversion of milk-sugar, and this circumstance entirely vitiated the process, since the same osazone would be produced both from cane-sugar and from milk-sugar. A far more hopeful plan would be to invert the cane-sugar by citric acid, as proposed by Messrs. Stokes and Bodmer some years since, as this reagent left the milk-sugar unchanged.

Mr. BODMER said that the method of inverting with citric acid was, he believed, one of the simplest available for the estimation of cane-sugar in milk. It was quite possible to prove whether a sample of milk contained, say, 0.5 per cent. of cane-sugar in about twenty minutes. The milk was coagulated in the way described in the paper (except that citric acid was used instead of acetic), made up with alkali, and titrated with Pavy's solution. Another portion of the same solution was inverted by boiling with citric acid, neutralized and titrated, the increase in reducing power being due to cane-sugar.

Mr. HEHNER thought that the use of acetic acid as a precipitant was a retrograde step. Although it removed by far the larger proportion of the albuminoids, it left behind nitrogenous substances of high rotatory power, the presence of which would affect the results.

He would like to ask Mr. Blyth if he had ever in actual practical work met with a milk containing any carbohydrate besides milk-sugar.

Mr. BEVAN inquired what was the smallest quantity of cane-sugar that could be accurately determined by the methods employed by Mr. Blyth. He believed that in Mr. Blyth's book it was stated that it was useless to attempt to determine cane-sugar unless it could be distinctly tasted.

The method of separating the albuminoids by means of acetic acid was in operation in the French municipal laboratories, although the details of its application were somewhat different. They used a ribbed filter, pouring in first 200 c.c. of very dilute acetic acid, and then from 10 to 20 c.c. of the milk. After a few minutes the tap at the bottom of the funnel was opened, and the filtrate allowed to run through. The sugar in the filtrate was estimated by means of copper solution, as it was too dilute for the polarimeter. The residue—allowed to dry spontaneously—was an excellent preparation for estimating the fat, the ribbed filter being placed in a Soxhlet and treated with ether.

Mr. CASSAL said he thought the only recorded case of a sample of milk adulterated with sugar and submitted officially to a public analyst was one given to the Society by Dr. Muter, who had determined the milk-sugar and added sugar.

In precipitating condensed milk—which was necessary in order to make an accurate determination of the fat—he had found that sulphate of copper gave very satisfactory results.

Mr. WYNTER BLYTH said he agreed with Mr. Richmond that mercuric nitrate, as a precipitant only, possessed certain advantages, but the filtrate could not be used for any other purpose, which was a great disadvantage. It might, of course, be possible to invert successfully in a filtrate in which a salt of mercury was present, but he thought it preferable that there should be as little foreign matter as possible in the filtrate. He had been disappointed with cheap polarimeters. His experience led him to think it worth while paying a little more, and getting a really serviceable instrument which could be relied upon.

He had never made use of invertase. As a matter of fact, this investigation was not at all complete, it having been interrupted by numerous engagements of one kind and another, but he ventured to hope that the discussion it had raised might be considered a useful outcome of the paper as it was.

He had not been aware of the fact stated by Mr. Allen of the solubility of the osazones in ether.

From Mr. Bodmer's remarks he imagined that the citric acid process would be very useful, although he had not as yet availed himself of it.

He did not think that less than about $\frac{1}{4}$ per cent. of cane-sugar could be certified to with absolute confidence. In reply to a further question by Mr. Bevan, he said that all milk samples passing through his hands were now tested with the polarimeter, but this had only been done for a very short time as a matter of routine, which would account for his being unable to say that he had ever met with a sample adulterated with cane-sugar in actual work.

The PRESIDENT said that they were much indebted to Mr. Blyth for this paper; anything which added to what was known regarding the relationship between the various constituents of milk was not only of interest to public analysts, but formed a substantial advance in scientific chemistry.

Papers were read on the following subjects: "A Comparison of the Organic Carbon and Nitrogen Results obtained by Dr. Frankland and the Companies' Analysts from the Waters supplied by the Metropolitan Water Companies," by W. C. Young; "Note on the Detection of Formalin in Milk Samples," by S. Rideal, D.Sc. The publication of these is unavoidably postponed.

NOTES ON CINNAMON.

BY BERNARD DYER, D.Sc., AND J. F. H. GILBARD.

(Read at the Meeting, February 6, 1895.)

ONE of us was lately informed that a certain firm of confectioners abroad, who did a heavy trade in walnuts, which they used in various forms for confectionery, had a large sale for their walnut-shells—in fact, that they sold them in London for more than they gave for the whole walnuts, the shells being used in adulterating ground cinnamon.

As, in addition to this statement, information to somewhat the same effect was made public, it appeared likely that ground cinnamon might receive the attention of food inspectors; and as it also appeared that little information was available about either cinnamon or ground walnuts, some typical samples were obtained from Messrs. Joseph Travers Sons, Limited, representing "cinnamon chips," "broken cinnamon," and three kinds of "quill cinnamon," two of ordinary and one of a "superior" variety. We examined these, and also a sample of finely-ground walnut-shells.

The microscopic detection of powdered walnut-shells in ground cinnamon is not difficult to a skilled observer; but an inexperienced microscopist might well be misled if he did not very carefully study both structures. Cinnamon-bark contains a large proportion of very hard sclerenchymatous tissue, the dense, internally thickened cells of which are, but for size, not wholly dissimilar from the sclerenchyma con-

stituting the harder portion of the walnut-shell. Any microscopical observation made with a view to the detection of walnut-shells should not be undertaken, therefore, without a careful independent study of the microscopical structure of cinnamon-bark itself, as well as of the supposed adulterant.

Assuming that the microscopical examination gives evidence of the presence of ground walnut-shells, the question of estimating the quantity present has to be met; and it is with a view to furnishing data for such purposes that the analyses were made which resulted in the following figures.

"Approximate Volatile Essential Oil" merely means the difference between the ether extract lightly dried on the top of the water-oven and the fixed ether extract obtained by complete drying inside the water-oven; the figure, in fact, being comparable to that given for "Approximate Volatile Essential Oil" in our previous paper on "Ginger."

"Fixed Ether Extract" is the non-volatile portion of the matter extracted by repeated exhaustion with ether until no more can be obtained.

"Alcoholic Extract after Ether" is the whole of the matter removable by prolonged exhaustion with alcohol in a Soxhlet apparatus after the removal of the ether extract.

"Fibre" is woody fibre obtained in the same way as in a feeding-stuff analysis.

	Moisture (Loss at 100° C.).	Approximate Volatile Essen- tial Oil.	Fixed Ether Extract.	Alcoholic Extract after Ether.	Total Ash less Sand	Ash soluble in Water.	Ash in- soluble in Water.	Fibre.	Nitro- gen.
Cinnamon chips ...	12.57	0.77	2.10	12.43	3.96	0.19	3.77	34.10	0.50
Broken cin- namon ...	11.33	1.53	2.30	12.90	4.80	0.10	4.70	34.33	0.54
Quill cinna- mon (a) ...	13.00	1.87	2.13	13.27	2.97	0.90	2.07	35.67	0.54
Quill cinna- mon (b) ...	12.50	1.77	1.87	11.00	4.40	0.63	3.77	34.27	0.42
Quill cinna- mon "su- perior" ...	12.65	1.93	2.30	13.23	5.00	0.50	4.50	32.90	0.54
Average of these five samples ...	12.41	1.57	2.14	12.57	4.22	0.46	2.76	34.25	0.51
Ground wal- nut-shells	9.97	0.27	1.60	3.67	0.87	0.37	0.50	47.67	0.20

The items "Volatile Oil," "Alcoholic Extract," "Insoluble Ash," and "Nitrogen" would all be obviously of assistance in calculating the percentages in a mixture of ground cinnamon and walnut-shells.

The fixed ether extract is not of much value for such purposes, and the soluble ash varies so greatly as to be of no use at all.

1.



3.

The woody fibre in cinnamon is pretty constant, and is appreciably less than in walnut-shells.

ADDENDUM.

The accompanying micro-photographs show the appearance, under high and low powers of the microscope, of bleached schlerenchymatous cells prepared by ourselves from both cinnamon and walnut shells.

No. 1, cinnamon under a low power; No. 2, ditto, more highly magnified; No. 3, walnut shells under a low power; No. 4, ditto, more highly magnified.

The photographs were very kindly made by Mr. A. Ashe, and have been reproduced by Messrs. Waterlow and Sons.

CRYSTALLIZED GLYCERIN.

(Meeting, April 3, 1895.)

Mr. Bevan exhibited some specimens of crystallized glycerin. He said that some time back a sample of glycerin had been submitted to him, which consisted of a mass of small crystals resembling soft sugar. He had found that by dropping a small quantity of the crystals into ordinary glycerin kept at a temperature of about 15° C., fresh crystals could be grown at a considerable rate.

DISCUSSION.

The Chairman (Mr. Otto Hehner) thought that, since commercial glycerin was not anhydrous, the glycerin above the crystals (unless the crystallized glycerin was a hydrate, which he believed was not the case) would become more and more dilute as the crystallization increased. It was remarkable that glycerin should crystallize out of an aqueous solution, since it was only anhydrous glycerin that was supposed to crystallize.* This seemed to suggest that glycerin was a crystalline substance in a state of supersaturation, the occasional upsetting of which showed the glycerin in its true state.

He would like to know whether an actual crystal was absolutely necessary to start the crystallization, or whether anything else would serve as a nucleus.

Mr. BEVAN said that the glycerin above the crystals, or mother-liquor, so to speak, was weaker than the original glycerin, and of course much weaker than the crystals. The case was different, however, from that, for instance, of water, which, when cooled down below its congealing point, crystallized in a solid mass the instant it was touched, whereas, in the case of glycerin, the process required a considerable time.

He had never been able to obtain crystallization except by starting with an actual crystal. Professor Dewar had obtained glycerin in a solid mass resembling glass, by cooling with the aid of solid carbonic acid and ether.

* Since speaking, I have analysed both crystals and mother-liquor. The former are pure anhydrous glycerin; the latter contains 6 per cent. of water.—O. H.

Mr. BODMER said that a sample of glycerin existed some years back in the dispensary at Guy's Hospital, which was absolutely solid. There was no mother-liquor at all, the material being exactly like a piece of ice.

THE COMPOSITION OF SOME ENGLISH CHEESES.

By W. CHATTAWAY, T. H. PEARMAIN, AND C. G. MOOR, M.A.

(Read at the Meeting, April 3, 1895.)

IN the July number of *THE ANALYST* of last year we published some figures representing the composition of several different kinds of cheese, and as we have recently been supplied with a series of specimens of English manufacture, we think the figures worth placing on record, particularly as they have all been made under proper scientific supervision and regulated conditions.

Some of them are imitations of foreign cheeses, and appeared to us to compare favourably with those made abroad.

With regard to the estimation of fat in cheese, after having paid some considerable attention to the question, we prefer the following process :

50 grammes of the cheese are ground up in a mortar with a fairly large quantity of sand. The powder so obtained is placed in a tall stoppered cylinder, and extracted by means of four successive portions of ether, using in all about 500 c.c. The ether washings are then made up to a definite volume, an aliquot portion taken, and the ether removed in the usual way. The residue of fat can then be subjected to further treatment in order to prove that it is true milk-fat.

When it is merely necessary to estimate the fat, this can be quickly and accurately done by means of the following modification of the Leffmann and Beam process for milk.

2.0 grammes of the cheese are taken, reduced to as fine a state of division as possible, then transferred to a small dish and treated on the water-bath with 30 c.c. of concentrated hydrochloric acid until solution is effected, and the fluid assumes a dark purplish colour. The mixture is now poured into a Leffmann-Beam bottle, the dish rinsed with the hydrochloric acid fusel-oil mixture into the bottle, and, finally, enough strong hot acid added to fill the bottle up to the mark. It is then centrifugated for about one minute. The Leffmann-Beam bottles are graduated so that ten divisions equal 1.0 per cent. by weight of fat on the 15.55 grammes of milk taken.

It follows, therefore, that the factor to be made use of in this case will be :

$$\frac{15.55}{2} = 7.7$$

With very little practice concordant readings are easily obtained, which agree very closely with the extraction process already explained.

We estimate the nitrogen by the Kjeldahl method, but find it essential to use a capacious distillation head in order to obviate the great inconvenience caused by the frothing which generally occurs when estimating the nitrogen in cheese by this process.

Name of Cheese.	Fat.	Water.	Ash.	Nitrogen.	Proteids N × 6.3.
Port de Salut	36.2	31.3	4.6	4.2	26.5
Caerphilly	30.4	24.8	3.4	5.9	37.2
Culommier	24.1	37.8	4.1	3.9	24.6
Cleveland	35.0	38.0	3.4	4.4	27.7
Cambridge	47.1	32.1	4.4	3.9	24.6
Gorgonzola	33.2	33.5	3.5	6.0	37.8
Double cream	68.1	14.0	1.2	3.2	20.1
Camembert	33.2	35.0	2.9	5.5	34.6
Gervais	69.3	15.8	0.6	3.0	18.9
Wensleydale	33.3	28.3	3.7	4.3	27.2
Cheddar	30.5	37.7	3.9	4.6	29.0
Stilton	34.6	25.0	4.1	4.5	28.4

DISCUSSION.

Mr. ALLEN drew attention to the large differences between the amount of fat in the various cheeses, remarking that it was at the same time rather striking that all the figures for fat, excepting for the Cheddar, were over 35 per cent. He believed that the authors had mentioned in a previous paper a Dutch cheese containing only 10 per cent. of fat. These differences emphasized very strongly the obstacles with which the fixing of standards was beset, and the difficulty of laying down limits which should ensure equal justice to the trade and to the consumer. It was well to keep a watch upon cheeses containing very small proportions of fat, although it might not be advisable at present that such should be absolutely condemned. It was very desirable that as complete data as possible should be placed on record regarding the composition of articles of food as arrived at by modern methods, and he thought the authors of the paper had done good work in this direction.

Dr. VOELCKER said that he should have expected to find a greater difference between the percentages of fat in Stilton and Cheddar cheese than the 4 per cent. indicated in the table. It was the practice in making Stilton cheese to add a considerable amount of cream, and this would result in an excess of fat over that in the Cheddar—which was an ordinary cheese, and not a cream cheese—of considerably more than 4 per cent.

Dr. RIDEAL said that as the amount of nitrogen as determined would probably in most of the cheeses indicate, not only proteids, but also ammonia and other decomposition-products, it was as well to calculate the exact relation between the nitrogen and the proteids for each cheese.

Mr. BEVAN said that in the case of the first three cheeses in the table, if the proteids were taken at $6\frac{1}{2}$ times the nitrogen, the figures added up to just under 100. In the case of Gorgonzola the sum of the figures came to 107.7, but an error of something over 1 per cent. in the nitrogen determination would account for this. He did not, of course, mean to imply that $6\frac{1}{2}$ was necessarily the right number, but it was interesting to note that the figures obtained by using it agreed very well in most of the cases.

The CHAIRMAN (Mr. Otto Hehner) pointed out that the authors had only dealt with cheeses of English make, and that the composition of the English-made imitations of foreign cheeses should not be taken as affording information as to the composition of the "genuine" articles.

Cheese analyses ought not to be expected to add up to 100, as multiplication of the nitrogen by $6\frac{1}{4}$ to obtain the proteids assumed that the nitrogen existed in the form of albuminoids only, which was not the case, especially in Gorgonzola and similar cheese, which contained a proportion of nitrogenous basic substances.

With regard to standards, in America the conclusion had been reached that at least 40 per cent. of the total solids in genuine cheese should consist of fat. Seeing that in normal milk the fat was about equal in amount to the albuminoids, one half of the total solids in cheese ought to be fat, although this was frequently not so, even in what would be called good cheese. In England, cheese made from skimmed milk, provided that it contained no fat other than milk fat, was passed as genuine, which was unfair to the manufacturers of whole milk cheese. In the case of milk, the sale of skimmed milk, when milk was asked for, was illegal, and skim-cheese ought to be on the same footing.

Mr. C. G. Moor said that they had hardly expected the nitrogen determination to escape criticism, as the separately determined constituents did not add up quite so close to 100% as might have been wished for. The factor 6.3 did not always answer, although he could not think that this was due to the formation of ammonia, as the samples were all perfectly fresh when examined.

The imitations of foreign cheeses had all been made under as nearly as possible the same conditions as prevailed abroad, from the same materials as the originals, and in the same proportions.

THE TESTING OF OILS WITH THE OLEO-REFRACTOMETER.

By THOMAS H. PEARMAIN.

(Read at the Meeting, April 3, 1895.)

Now that the oleo-refractometer, as devised by Messrs. Jean and Amagat, is coming so largely into use in commercial work, the following table, which gives a summary of the results of the examination of 240 samples of oil, may be of interest.

I find much better results are obtained with this instrument when the temperature is falling, especially in the case of butter and margarine, which, owing to their higher melting-point, have to be observed at the higher temperature, namely, 45° C.

If the sample of oil under examination is very acid, it is necessary to remove the free fatty acid by shaking the oil in a separator with hot alcohol, after which treatment the oil is dried at 105° C.

In testing oils which give a high deviation, as the fish oils, for instance, it is desirable when making the observation to dilute them with their own volume of lard-oil, whereby a much sharper shadow is obtained. The figure so obtained is, of course, doubled to obtain the proper refraction number of the oil.

It is to be regretted that the oleo-refractometer is not a more satisfactory test in the case of butter and margarine. My results confirm those of G. H. Ellinger (*THE ANALYST*, 1891, p. 197), who tested 510 samples of Danish butter, and found the greatest difference of deviation to be from 35° - 23° when examined in this instrument at 45° C.

Temperature 22° C.	Number of Samples Examined.	Deviation.	Highest.	Lowest.	Average.	Temperature 22° C.	Number of Samples Examined.	Deviation.	Highest.	Lowest.	Average.
Almond ...	8	+	10.5	8.0	9.5	Poppy-seed ...	3	+	35.0	30.0	33.0
Arachis (Earth Nut) ...	5	+	7.0	5.0	6.0	Rape ...	8	+	20.0	16.0	17.5
Bottlenose ...	1	+	50.0	50.0	50.0	Ravison ...	2	+	24.0	20.0	22.0
Cabbage Seed...	1	+	15.0	15.0	15.0	Seal ...	2	+	36.0	30.0	33.0
Castor ...	8	+	42.0	39.0	40.0	Sesamé ...	5	+	17.0	13.0	15.5
Cod-liver ...	8	+	46.0	40.0	44.0	Shark ...	3	+	35.0	29.0	31.0
Cotton-seed (Crude) ...	3	+	17.0	16.0	16.5	Sunflower ...	1	+	35.0	35.0	35.0
Cotton-seed (Refined) ...	6	+	23.0	17.0	21.5	Tallow Oil ...	2	-	5.0	1.0	3.0
Hempseed ...	4	+	37.5	34.0	35.5	Tea ...	1	+	3.0	8.0	8.0
Japan-wood Oil	1	+	75.0	75.0	75.0	Whale ...	2	+	48.0	42.0	45.0
Lard Oil ...	6	-	1.0	0.0	0.0	Oleic Acid ...	3	-	33.0	29.0	32.0
Linseed (Crude)	3	+	52.0	48.0	50.0	Temperature 45° C.					
Linseed (Re- fined) ...	5	+	54.0	50.0	52.5						
Neat's Foot ...	2	-	3.0	1.0	2.0	Butter ...	15	-	34.0	25.0	30.0
Niger-seed ...	2	+	30.0	26.0	28.0	Margarine ...	7	-	18.0	13.0	15.0
Olive ...	105	+	3.5	1.0	2.0	Lard ...	10	-	14.0	8.0	10.5
Peach Kernel...	2	+	11.5	7.5	9.5	Tallow ...	6	-	18.0	15.0	16.0
Pilchard ...	2	+	36.0	32.0	34.0	Paraffin (Soft)...	2	+	58.5	54.0	56.0

DISCUSSION.

Mr. BEVAN said that it was his practice to examine with the oleo-refractometer every sample of butter passing through his hands, and he had never found it lead to a mistake. Its working, however, depended to some extent upon the personal element, as well as upon the exact position of the prism, and there might be differences between the results of different observers using different instruments.

He suggested a mixture of glycerine and water as a substitute for the standardizing oil supplied with the instrument. It would be much cheaper, and would be free from the disadvantage of being only obtainable in Paris.

He took exception to the use of the term "refractive index," which he thought might convey misleading ideas.

Mr. ALLEN agreed with Mr. Bevan as to the usefulness of the instrument. He thought, however, that the price charged for it was a great deal too high. He had reason to believe that the angle of the prism was not strictly the same in all instruments, and this would account for some of the discrepancies to be found among the results of different operators. But a sample of lard, which showed $41\frac{1}{2}^{\circ}$ in his own instrument, gave 6° in Mr. Bevan's and 11° in that of another well-known analyst, all these observations being made by Mr. Allen personally. This state of things was a disgrace to the manufacturers of the instrument.

The CHAIRMAN questioned whether the oleo-refractometer afforded any more information than could be obtained from the iodine absorption test. The figures given in the paper seemed to him to show that oils of high iodine absorption also had a high refractive power, and that the latter stood in direct proportion to the iodine absorption. If this was really the case, he thought the iodine absorption test was much to be preferred.

NOTE ON THE DETECTION OF COTTON-OIL IN LARD.

By E. B. KENRICK.

VARIOUS conflicting statements have been made regarding the absorption-spectrum of cotton-seed oil, some writers affirming that the oil gives a banded spectrum, others asserting the contrary. Some years ago, when much attention was being paid by analysts to the detection of cotton-seed oil in lard, I examined spectroscopically a number of samples of the oil. The samples included specimens obtained from American and Canadian lard manufacturers, as well as the pale yellow varieties sold as cheap salad-oil. Marked differences were observed even among samples resembling each other in outward appearance; all the samples with one exception showed, when first obtained, in layers an inch or less in thickness, a banded spectrum. In most instances the bands gradually disappeared on keeping. A sample of crude-oil, however, still showed the chlorophyll band in the red at the end of six years. The sample referred to as giving no banded spectrum was a sample of "white" oil from a Chicago lard manufacturer. It appears to be the kind commonly employed in lard compounds. When examined in thicknesses of two feet, the oil had a yellow colour, and cut off all light more refrangible than the green, but no traces of bands appeared. During the last six years I have frequently examined the absorption-spectrum of compound lards, in the hope of detecting vegetable oils by this method, but until recently I have always obtained negative results. I have now come across samples which give the chief chlorophyll band in the red, when examined in thicknesses of two or three inches.

The delicacy of the spectroscopic test will depend, of course, on the nature of the cotton-seed oil used, and the test is quite worthless for quantitative purposes. Though no conclusion as to the presence of cotton-seed oil can be drawn from negative results, on the other hand, in cases where the less highly-refined oil has been employed, a few per cents. of the oil may be detected by passing the light through a sufficiently long column of the melted fat.

St. John's College, Winnipeg.

A Modification of Renard's Process for the Detection of Arachis Oil.
H. Kreis. (*Chem. Zeit.*, 1895, xix., 451-452.)—The detection of arachis oil is usually effected by means of the characteristic properties of arachidic acid. The separation of the soluble from the insoluble lead salts of the total fatty acids, by taking advantage of the difference of their solubility in ether, presents some difficulty on account of the pasty consistency which they possess. The author has mitigated this

objection by precipitating the solution of the fatty acid with an alcoholic solution of lead acetate instead of the usual aqueous liquid. The process thus worked out may be described as follows: 20 grammes of the oil are saponified with 10 c.c. of caustic soda solution (40 per cent.) and 50 c.c. of alcohol (90 per cent.). After distilling off the alcohol, the fatty acids are thrown down with hydrochloric acid, washed with hot water, and dissolved in 100 c.c. of alcohol of the strength given above. The solution is cooled in ice water, and to it is added, with constant stirring, a solution of 15 grammes of lead acetate in 150 c.c. of alcohol (90 per cent.). The precipitate, after standing for two hours, is filtered through cotton-wool by the aid of a pump, and extracted for six hours with ether. The residue is then placed in a porcelain dish, and boiled with 250 c.c. of hydrochloric acid (5 per cent.) until the fatty acids swim upon the surface as a clear oily layer. The acids thus obtained are washed two or three times with hot water to remove lead chloride, dried by pressing between filter paper, dissolved by gentle warming in 100 c.c. of alcohol (90 per cent.), and the solution cooled to 15° C. for several hours, by which process separation of arachidic acid takes place, the identification of which is effected in the usual manner.

When no special economy in ether is requisite, the process may be simplified as follows: The fatty acids obtained from 20 grammes of oil are dissolved in 300 c.c. of ether, and treated at the temperature with 150 c.c. of the alcoholic solution of lead acetate mentioned above. Lead oleate remains in solution, and the precipitate which forms after a few hours consists almost wholly of the lead salts of solid fatty acids. The precipitate is collected, washed with ether, and identified as in the previous case.

B. B.

On the Examination of Linseed Oil and Boiled Linseed Oil. F. Filsinger. (*Zeit. für angewand. Chemie*, 1895, pp. 158, 159.)—The author here replies to Dr. Amsel's criticisms on his method of examining oils with the polarimeter (see *ANALYST*, xx., p. 70). The dark colour of the chloroform solution of boiled oils is not of much account, since with the apparatus of Schmidt and Haensch a colourless solution is not essential. No difficulty in taking the observation will be experienced if the solution of the boiled oil be first filtered, and in many cases it is so clear that this is not required.

It is true that mineral oil is optically inactive; but in the first place the saponification figure will detect it, and in the second place any extensive adulteration with it is unlikely, since a small addition would yield no profit, while a larger one would altogether prevent the oil from drying. There is, therefore, but little danger of its leading to erroneous conclusions with the polarimeter. It is also doubtful whether rape-oil occurs in any quantity in linseed-oils, since it is dearer than the latter. For this reason the author doubts the interpretation of the results with oil No. 10 in Dr. Amsel's second table.

C. A. M.

Estimation of Dust in Air. Karl Arens. (*Arch. Hyg.*, xxi., pp. 325-328.)—A tube 8 to 10 centimetres in length is drawn out at one end and connected with an aspirating apparatus on the principle of the bellows. A loosely-packed layer of cotton-wool 3 to 4 centimetres thick is introduced, and 200 litres of

air drawn through by means of the aspirator during half to three-quarters of an hour. Before connecting, the dry tube and wool are weighed, and after the air has been drawn through they are allowed to stand over concentrated H_2SO_4 for twenty-four hours, and again weighed, the increase in weight being calculated as dust. The following table gives the quantities found by the author in a cubic metre of air from different sources :

Milligrammes.				Milligrammes.			
Dwelling-room				Iron foundry (15 to 20 workmen)			28.0
Laboratory	1.4	" "	(not previously used)		1.5
Schoolroom	10.0	" "	(few workmen)		12.0
Horse-hair factory	17.0	" "	(during interval)		8.0
Sago factory	17 and 15	Snuff factory	72.0
Woollen factory, picking-room	7.0	" "	before grinding		16.0
" " cutting-room	20.0	Cement works (during work)			224.0
Flour mill	22 and 28	" "	(in an interval)		130.0

C. A. M.

The Action of Magnesia Mixture on Glass. L. L. De Koninck. (*Chem. Zeit.*, 1895, xix., pp. 450, 451.)—The formation of a deposit in magnesia mixture prepared for the determination of phosphoric acid is a familiar phenomenon, no matter what care be taken to ensure purity of the constituent substances and perfect clarity of the solution resulting from their admixture. A white flocculent deposit always appears in the reagent bottles used for containing this particular solution. The author has collected and examined the substance thus separated, and finds that it consists of small white iridescent scales saponaceous to the touch and strongly resembling powdered talc. On drying at 100°C . the substance loses 13.5—18 per cent. water, but retains its original aspect, becoming, however, excessively hygroscopic. A sample analysed has the following composition :

	Per cent.
Water at 100°C .	13.43
Water above 100°C .	17.86
Silica	32.99
Magnesia	34.80
Alumina and ferric oxide	0.87

99.95

The substance becomes greyish on ignition, this being due to its containing a small quantity of iron. The ignited substance is no longer hygroscopic.

The author has endeavoured to determine whether the composition of the glass of the vessels in which the magnesia mixture is kept appreciably influences the character of the precipitate formed. He finds that whether the glass have soda or potash as its characteristic alkali, it exercises no influence on the composition of the precipitate, which is sensibly similar in all cases. The quantity of the precipitate formed, however, varies notably with the nature of the glass. Thus, bottles of Thüringian glass are much more rapidly and severely attacked than those made of Bohemian glass or that of Stas. In the case of the last-named material the quantity of deposit produced is quite small. For example, a bottle exposing a surface

of 90 sq. cm. caused the formation of only 0.084 grammes of precipitate during a period of twenty-seven months. B. B.

On the Estimation of Bromine in the Presence of Chlorine. W. Wense. (*Zeit. für angewand. Chemie*, 1895, Heft i., pp. 13-14.)—For the purpose of rapidly determining small quantities of bromine in the presence of much chlorine, the author proposes an indirect process. It is based on the fact that when a mixture of Cl and Br is passed through a solution of KI an equivalent of I is liberated, the quantity of which can be determined by titration. And, further, by the liberation of the I by the Br and Cl, there will be a diminution in weight in the salt in the KI solution. The amount of this can be ascertained by evaporating part of the solution. From these two data the quantities of Br and Cl can be determined.

In making the estimation the salt solution to be examined is distilled with rather more chlorine water than is required to liberate all the Br. Care must be taken that neither the salt solution nor the chlorine water contain free acid. The Cl and Br distilling over are conducted through KI solution containing a known weight of KI, any of the usual apparatus being used for the purpose. The distillation is stopped when all the Br has passed over, a point which is determined by experience. With some salt solutions it happens more speedily than with others. Thus, $MgCl_2$ solutions retain free Cl and Br much more than solutions of the alkaline chlorides. The KI solution is next made up to a definite volume, and the amount of I liberated determined by titration in an aliquot part. A second aliquot part is evaporated to dryness on the water-bath in a porcelain dish, and the residue heated for half an hour at from 160° to 180° in an air-chamber. The temperature must not reach 200° , as, according to Petersson (*Zeit. Anal. Ch.*, 1871, 362), KI then becomes partially decomposed. This will give the loss in weight, caused by the substitution of Br and Cl for I in the KI.

In calculating the results two equations are used, which are obtained from the following data :

1 mg. Br acting on the KI causes a loss of weight in the latter of 0.5866 mg.

1 mg. Cl acting on the KI causes a loss of weight in the latter of 2.5773 mg.

If I = the weight of liberated I found by titration,
 D = loss of weight in the KI,
 $\left. \begin{array}{l} c = \text{quantity of Cl} \\ b = \text{quantity of Br} \end{array} \right\} \text{absorbed by the KI solution,}$

then

$$(1) \quad I = c + b + d,$$

$$(2) \quad D = 0.5866b + 2.5773c;$$

and from these

$$c = 0.797 D - 0.295 I,$$

$$b = 1.295 I - 1.797 D.$$

Example.—A solution was made up containing 338.5 mg. KBr, 20 grammes NaCl, and 80 c.c. H_2O , and this gave for I 1175.3 mg. and for D 721 mg., from which was calculated the amount of Br = 226.4 mg., instead of the theoretical 227.3 mg.

C. A. M.

Cactus Alkaloid.—Lewin finds (*Ber. Deutsch. Bot. Gesell.*, 1894, pp. 283-290; through *R. Micro. Soc. Journ.*, 1895, p. 191) in *Anhalonium Lewinii* and some other species of Cactaceæ, a poisonous alkaloid resembling strychnine in its properties.

F. H. P. C.

The Volumetric Analysis of Chloroplatinates; the Determination of Potassium, Ammonium, Nitrogen, and Platinum. L. L. de Koninck. (*Chem. Zeit.*, xix., 1895, 301, 302.)—All methods which have been devised for the determination of potassium volumetrically are necessarily indirect, the element (usually chlorine) which is combined with the potassium being actually estimated. Thus a process exists consisting in obtaining the potassium as the chloroplatinate, igniting this salt with a reducing agent, *e.g.*, hydrogen or oxalic acid, and titrating the potassium chloride, extracted from the residual metallic platinum, by means of silver nitrate in the ordinary way. A disadvantage of this process is that the exactitude which might be attained by taking advantage of the high molecular weight of potassium chloroplatinate is sacrificed in the course of ignition, all chlorine other than that sufficient to form KCl being dissipated. Mohr has endeavoured to meet this objection by igniting with sodium oxalate instead of oxalic acid, but the method is tedious, involving neutralization of the mass after heating and before titration. The author, some time ago, published a method (*Rev. Univ. des Mines*, 1881, 9), in which the chloroplatinate is dissolved in boiling water and reduced by means of magnesium ribbon. The process is not free from error, inasmuch as an insoluble platinum compound appears to be formed, together, it appears, with some magnesium oxychloride. The substitution of magnesium powder for ribbon gives better results, but it must be noted that commercial magnesium in both forms contains an appreciable amount of chlorine which is not due to mere surface contamination, but exists in the body of the metal. In consequence of this, not only must the magnesium employed be weighed, but also care must be taken to ascertain the evenness of the distribution of the chlorine in the metal. In consequence of these drawbacks, the author has adopted the use of formic acid (already recommended by Corenwinder and Contamine, *Comptes Rendus*, 1879, lxxxix., 907), as a reducing agent. The chloroplatinate is filtered and washed in the usual manner, dissolved in boiling water, and decomposed by calcium formate, freed from chlorine by re-crystallization. The solution is heated until the platinum has fully separated and the supernatant liquor is colourless; it is neutralized with a small quantity of calcium carbonate (prepared from the nitrate), filtered, and the chlorine determined by titration with silver nitrate in the customary way. The process is equally applicable to the ammonium salt. It can also serve for the determination of nitrogen in all cases where that element can be obtained quantitatively as ammonia, and will suffice for the estimation of the platinum on the same portion as that taken for the chlorine, all that is needful being a filtration previous to neutralization. Should bromine as well as chlorine be present, no error in the determination of the potassium or ammonium will be introduced, as the process is volumetric and valid for both halogens. Chloroplatinates can be analysed in precisely the same way. Reduction of platinum by a formate is useful

in removing excess of platinum chloride from a solution containing sodium chloride from which potassium has been separated. In this case ammonium formate (best made *in situ*) is of course used. B. B.

Method of Determining Chromium in Chrome Ore. Edmund Clark. (*Journ. Amer. Chem. Soc.*, 1895, xvii., pp. 327-330).—About .5 gramme of the finely powdered ore is fused with 25 grammes of KHSO_4 in a platinum crucible with a capacity of not less than 50 c.c. About forty minutes are required for the complete decomposition of the ore, and the greatest care is necessary to insure complete fusion, and to prevent loss. The fused mass is dissolved in a platinum evaporating dish by boiling gently with 35 c.c. HCl (sp. gr. 1.20) and 25 c.c. of water. What is left in the crucible is also dissolved and added to the rest, and the liquid is heated on the water bath in a beaker till nothing but silica remains undissolved. This is filtered off and weighed.

The filtrate is rendered slightly alkaline with ammonia, the hydroxides of chromium, aluminum and iron filtered off, dissolved in HCl , reprecipitated with ammonia, and collected on the same filter. The precipitate is washed with hot water, and lime and magnesia determined in the united filtrates by any good method.

The filter containing the mixed hydroxides is transferred to a $4\frac{1}{2}$ -inch porcelain evaporating dish, and oxidized by warming very cautiously with 50 c.c. of HNO_3 (sp. gr. 1.42), with the addition from time to time of small quantities of KClO_3 . The chromium now exists as chromic acid.

The solution is transferred to a 12-oz. beaker, made up to about 150 c.c. with water, and the iron and alumina precipitated with ammonia. The precipitate is filtered, dissolved in dilute HNO_3 , reprecipitated, collected and washed on the same filter, and the iron and alumina determined by any good method.

The ammoniacal solution containing the chromium as ammonium chromate is acidified with HCl , and sufficient sulphurous acid water added to completely reduce the chromium, which, after the SO_2 has been completely removed, is precipitated with ammonia, and weighed as Cr_2O_3 .

Another method of determining the chromium is to moderately acidify the ammonium chromate solution with acetic acid, add an excess of clear plumbic acetate solution, allow the precipitate to settle for several hours, collect on a weighed filter, and wash with cold water. The filter is dried and weighed, and the difference in weight gives the weight of the plumbic chromate, from which the amount of chromic oxide may be calculated. C. A. M.

On a New Alkaloid in Coffee. Pietro Paladino. (*Gazzetta Chimica Italiana*, xxv., 1895, pp. 104-110.)—The author has found a new alkaloid in coffee which has physical and physiological properties quite different to those of caffeine. To this alkaloid he has given the name of "Coffearine." To obtain it the finely divided coffee is boiled with ten times its weight of water rendered alkaline with milk of lime, the boiling being repeated with successive quantities of water until the coffee is completely exhausted. The liquid strained off is filtered through linen, and basic

lead acetate added until the precipitation is complete. The precipitate is filtered off, the excess of lead in the filtrate removed by precipitation with H_2SO_4 , and the clear filtrate, after being concentrated, completely exhausted with chloroform in order to extract all the caffeine.

The liquid from which the caffeine has been removed is then heated on the water bath with an excess of H_2SO_4 in order to expel all the acetic acid. The syrupy mass is taken up with water, the insoluble matter removed by filtration, and the liquid decolorized by heating on the water bath with animal charcoal.

The solution is diluted with much water, and when cold, Dragendorff's reagent is added with constant shaking, until the flocculent precipitate becomes crystalline. The precipitate is filtered off, washed, and decomposed by warming with H_2SO_4 . After filtration, the liquid is warmed on the water bath, neutralized with PbCO_3 , and again filtered. The precipitation with Dragendorff's reagent is repeated several times until the precipitate of the double iodide of the alkaloid and bismuth is perfectly crystalline.

Oxide of silver is added to the clear liquid resulting from the decomposition of the double iodide, and after filtration the hydrochloride of the alkaloid is precipitated with HCl .

The alkaloid gives no precipitate with picric acid or mercuric chloride. With tannic acid it gives a white flocculent precipitate, and with platinic chloride a yellow crystalline one.

The mean results of the analysis were :

	Found.	Calculated for Formula.
		$\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_4$
C.	60.64	60.87
H.	5.74	5.80
N.	10.10	10.14

On frogs coffearine acts as a narcotic poison, 0.2 gramme proving fatal. On the other hand, as much as 0.8 gramme only produced a slight torpor in a medium-sized mouse.

C. A. M.

The Estimation of Paraffin in Crude Anthracene. Fr. Heusler and Jos. Herde. (*Zeit. für angewand. Chem.*, 1895, p. 253.)—The method proposed depends on the fact that, with the exception of paraffin, all the constituents of crude anthracene are soluble in fuming nitric acid. About 2 grammes of the crude substance are weighed into a 150 c.c. flask, and 25 c.c. of fuming nitric acid added—at first drop by drop—while the flask is cooled with ice water. After the anthracene has been completely digested with the acid, the flask is warmed on the water bath until the paraffin melts. On cooling, the paraffin is filtered off on asbestos, thoroughly washed with fuming HNO_3 , and afterwards with water until no longer acid. It is then washed with alcohol and warm ether into a reagent cylinder. The alcohol is poured into a weighed porcelain dish and evaporated on the water bath. Then the ethereal solution is added, the ether evaporated, and the residue of paraffin dried for half an hour at from 105° to 110° and weighed.

C. A. M.

Note on the Determination of Zinc. P. W. Shimer. (*Journ. Amer. Chem. Soc.*, xvii., 1895, pp. 310-312.)—In estimating zinc in ores also containing manganese, it is usual to separate the former by precipitating it as sulphide in a solution strongly acidified with acetic acid. When the manganese is high it is necessary to dissolve this precipitate and reprecipitate it. To avoid this the following method, which gives results agreeing closely with those obtained by the usual methods, is proposed.

About .8 gramme of the zinc ore is dissolved in HCl. When any insoluble manganese or zinc spinel is present, a fusion must be made. The HCl solution is evaporated to dryness, redissolved, and the silica filtered off, if this is to be determined. Where the silica is not required it need not be filtered off. Redissolve in HNO_3 (1:20), and evaporate to moist dryness. Add 100 c.c. of strong HNO_3 , and precipitate the manganese as dioxide by KClO_3 , as in Ford's method for manganese in iron and steel. Filter through asbestos by means of the filter-pump, and wash with strong HNO_3 , followed by cold water. The manganese in the precipitate may then be determined by dissolving it in standard ferrous sulphate and titrating the excess of ferrous sulphate with standard permanganate.

The filtrate is evaporated to dryness in a beaker, a little HCl added and again evaporated to moist dryness. On this solution the usual double basic acetate precipitation is made. The united filtrates are evaporated down to about 300 c.c., heated to boiling, removed from the flame, and a rapid current of H_2S passed through for thirty minutes. The zinc sulphide is filtered off, dissolved in HCl, and the zinc precipitated preferably as zinc ammonium phosphate. The precipitate, separated from the paper, is ignited cautiously at a low red heat, and weighed as zinc pyrophosphate. If calcium is to be determined in the filtrate from the zinc sulphide, the calcium oxalate must be dissolved and reprecipitated, since the first precipitate is sure to contain considerable quantities of alkaline chlorides.

C. A. M.

Standardization of Sulphuric Acid. F. S. Shiver. (*Journ. Amer. Chem. Soc.*, xvii., 1895, pp. 351-354.)—In 1892, M. Weinig* proposed a method for standardizing sulphuric acid, in which ammonia in slight excess was added to a measured portion of the acid, the solution evaporated to dryness, and the residue dried at 115° to 120° , and weighed as ammonium sulphate. The author finds that there is a slight decomposition on evaporating an aqueous solution of ammonium sulphate to dryness. To avoid this he moistens the residue with strong ammonia water, dries on the water bath, and afterwards at 110° to 120° , till the weight is constant. The results obtained by this method agree closely with those obtained by precipitation of the sulphuric acid as barium sulphate.

C. A. M.

* *Zeit. für angewand. Chem.*, 1892, pp. 204, 205; *Analyst*, xvii., 90.

CORRESPONDENCE.

To the Editors of THE ANALYST.

Medical School, Caxton Street, Westminster,
May 27, 1895.

SIRS,—I should be greatly obliged if you could find room in the next issue of *THE ANALYST* for the inclosed report, which I sent to the Medical Department of the Local Government Board on November 7, 1887. A comparison of the report on the four waters from Mountain Ash, with the remarks thereon made by Dr. Thresh in his paper (*ANALYST* for May, p. 104, remark 17), gives an instructive illustration of the manner in which advocates of the biological examination of water deal with chemical analysis.

I can only express a hope that the other remarks of Dr. Thresh in the same paper are more fair and to the point.—Yours, etc.,

A. DUPRÉ.

[COPY.]

The waters are all soft, and leave but little dry residue. The chemical character is almost identical, as, with one or two exceptions, all the variations observed are within the limits of experimental error. Taken separately, each one of the waters must be pronounced as very pure, and free from any indications of sewage pollution.

Looking at them, however, as all coming from the same general source, there are indications pointing to a slight degree of pollution in some of the waters.

Thus, contrasting No. I. with No. IA., it will be seen that the latter shows a little more dry residue, a minute trace (certainly a very minute trace) of ammonia not found in No. I., and a distinct increase in the proportion of phosphoric acid. In a similar manner, II. and IIA. are slightly less pure than No. I.

The nature of the deposits, however, furnishes far more marked difference. Thus, the deposit from No. I. consists almost entirely of mineral matter, oxide of iron chiefly, and contains neither fungoid growths nor living organisms.

The deposit from No. IA. contains particles of decayed wood, vegetable fibres, and numerous large animalculæ. The deposit from No. II. shows numerous starch grains (potato?), fungoid growths, vegetable fibres, the remains of some insect, some large and some small animalculæ. No. IIA. contains an appreciable amount of deposit, consisting mainly of oxide of iron and clay, but contains also fungoid growths, some naviculæ, and a few larger animalculæ.

Taking all points into consideration, No. I. is the most pure of the samples, while the remaining three samples show signs of pollution, indicated partly by their chemical character, more so by the character of their deposits.

(Signed) A. DUPRÉ.

November 7, 1887.

APPOINTMENT.

MR. LEO TAYLOR has been appointed Public Analyst for Hackney, in succession to the late Dr. Tripe.

THE ANALYST.

JULY, 1895.

Obituary.

DR. WILLIAM MORGAN, OF SWANSEA.

WE deeply regret to have to announce the death, which occurred on June 19 from Bright's disease, of Dr. W. Morgan. Dr. Morgan was the Welsh public analyst. He acted for the counties of Brecon, Carmarthen, Glamorgan, Monmouth, and Pembroke, and the boroughs of Swansea, Neath, and Carmarthen. We have lost by his death a most worthy man and an active and able public analyst.

Dr. Morgan was always proud to relate how he commenced life in the humble position of carpenter's apprentice in Carmarthen. Whenever he could, he attended lectures on science and art. In August, 1864, Mr. Morgan removed to Swansea, still working as a carpenter. Soon he found an opportunity of entering the Burrows Spelter Works as assistant chemist, and in 1864 he was appointed assistant chemist at Messrs. Vivian's great smelting works. By 1870 he had saved a small sum of money. He gave up his appointment with Messrs. Vivian, and proceeded to Giessen on the Lahn, the little German university which had for more than a quarter of a century been famous as the home of the great Liebig. Liebig himself had before 1870 left Giessen for Munich, and Morgan worked mainly under Professor Will. Here for two years he studied physiology, mineralogy, and chemistry, and at the end of that time obtained his Doctor's degree, *summa cum laude*. In 1873 he returned to Swansea and opened a laboratory. In the early part of 1874 he was appointed public analyst for Swansea. Other appointments rapidly followed. In 1884 he removed to more commodious premises in Nelson Terrace, and he gradually added to these, until the establishment in Nelson Terrace occupied, as it does at the present time, a large block of buildings, and arranged both for analytical work and for the teaching of students. Probably no better and more commodious laboratory exists in the country, and many a college might be proud to possess it. Excellent collections of books, of instruments, and of specimens are accumulated. Dr. Morgan, as an excellent German linguist, kept continually in touch with Continental literature, and all the newest and best books were eagerly obtained by him. In the construction of the laboratory his old training as carpenter stood Dr. Morgan in good stead. His analytical practice, apart from the work of the Food Act, was most extensive, and large numbers of spelters, copper-ores, and metallic products of all kinds passed through his hands.

He was highly respected by his fellow citizens, and occupied for many years the position of chairman of the Swansea School Board.

During Dr. Morgan's last illness Mr. Seyler, his chief assistant, was appointed interim analyst for several of the districts for which Dr. Morgan acted officially.

O. H.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday evening, June 5. In the absence of the President, Mr. Otto Hehner took the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election: As members—Robert William Atkinson, B.Sc. (London), F.I.C., Consulting Analyst, 44, London Square, Cardiff; J. Walter Leather, Ph.D., F.I.C., Government Agricultural Chemist, India, Dehra Dun, N.W.P., India. As associates—Herbert Burgess, assistant to Professor Graham; and T. H. P. Heriot, assistant to Mr. R. H. Harland.

Mr. Mitchell read the following paper:

A NEW THERMAL METHOD FOR THE EXAMINATION OF OILS.

By OTTO HEHNER, F.I.C., AND C. A. MITCHELL, B.A., A.I.C.

ONE of the earliest quantitative methods used in the examination of fats and oils is that of Maumené, in which the rise of temperature due to the interaction of oil and sulphuric acid is measured. Although practically very useful data can be obtained by the method, especially when employed in the more refined form introduced by Thomson and Ballantyne and others, yet it appears unlikely to afford any exact insight into the constitution of any particular oil, the rise of temperature being due to a great variety of causes. Thus the sulphuric acid causes hydrolysis of the glyceride, oxidation and sulphonation of the fatty acids, besides sulphonation of the glycerine, the oxidation not stopping short at any well-defined point, but often proceeding to the complete disruption of the molecule.

It is certain that the amount of heat evolved stands in some form of close relation to the degree of unsaturation of the fatty acid, for generally an oil consisting mainly of olein shows a less rise of temperature than a semi-drying oil containing linolein, and this, again, than drying oils containing a glyceride of linolenic acid. As the degree of unsaturation is now more or less accurately measurable by the iodine absorption, it follows broadly that the higher the iodine absorption the higher the Maumené figure. That this is the case when different specimens of the same oil are examined by the same observer is illustrated by the following table.

G. F. Tennille (*Jour. Amer. Chem. Soc.*, 1895, pp. 33-41; *Abts. Analyst*, 1895, p. 63) gives the Hübl and Maumené figures for nine samples of lard. When the

Maumené figure is multiplied by the factor 1.748, a number corresponding fairly closely with the Hübl figure is obtained :

	Maumené Figure.		Hübl No.		Calculated I. No.
1.	30.5	...	54.5	...	53.31
2.	31.8	...	55.6	...	55.59
3.	34.8	...	58.6	...	60.73
4.	30.0	...	53.7	...	52.44
5.	31.5	...	55.4	...	55.06
6.	31.7	...	56.1	...	55.41
7.	25.5	...	47.7	...	44.57
8.	31.5	...	51.7	...	55.06
9.	29.5	...	52.0	...	51.57

Similarly from eleven olive-oil analyses published by Lengfeld and Paparelli (*Rev. Inter. des Falsifications*, v., 1892, p. 98; *Abst. Jour. Soc. Chem. Ind.*), the Maumené number multiplied by 2.1837 also gives an approximation to the iodine number :

	Maumené Figure.		Hübl No.		Calculated I. No.
1.	37	...	80.80	...	80.80
2.	35	...	77.28	...	76.53
3.	39.5	...	87.35	...	86.26
4.	37.5	...	83.35	...	81.99
5.	41	...	88.68	...	89.53
6.	38	...	81.45	...	83.08
7.	36	...	79.50	...	78.61
8.	34.5	...	79.53	...	75.44
9.	33.5	...	78.42	...	73.26
10.	36.5	...	85.44	...	79.71

The samples are all stated to be undoubtedly pure Californian oils.

It will be seen that while the factor for the lards is 1.748, for the olive-oils it is 2.1837. Whether this is due to difference in manipulation or strength of acid, or whether the more unsaturated olive-oil evolves proportionately less heat than the lards, it is impossible to say from these figures.

That the same factor does not hold good for different oils, although analysed by one observer, is evident from the carefully determined figures of De Negri and Fabris (*Zeit. Anal. Chem.*, 1894, 547-574); the relation between the Maumené and Hübl figures calculated from the mean data of 203 samples of olive-oil is as 1 to 2.314. Applying this factor to other oils also analysed by De Negri and Fabris, we obtain the following figures :

	Maumené Figure.		Hübl No.		Calculated I. No.
Olive-oil (203 samples)	35	...	81	...	81
Earthnut	49	...	95 - 95.4	...	113.4
Hazelnut	35- 36	...	86.2- 86.8	...	81- 83.3
Almond	51- 52	...	93 - 95.4	...	118- 120
Rape	92- 95	...	108 -108.8	...	213- 220
Hempseed	95- 96	...	157.5	...	220- 222
Maize	86	...	111.2-112.6	...	199
Cotton seed	50- 53	...	106.9-110	...	115.7-122.6
Linseed	122-126	...	158.7-159.78	...	282 -291.5

It is obvious from these numbers that there is nothing like constant relation between the Hübl and Maumené numbers in the above cases.

Even when the specific temperature reaction as proposed by Thomson and Ballantyne, whereby differences in the strength of the sulphuric acid used are compensated, is taken as the basis of calculation, no one factor can be found equally applicable to various kinds of oils.

C. Fawsitt (*Journ. Soc. Chem. Ind.*, 1888, p. 552) has also attempted to utilize the heat evolved by sulphur chloride (S_2Cl_2). The reaction in this case is probably less complicated than in the case of sulphuric acid, but is still too involved to allow of definite conclusions being drawn from the thermic data. Thus, S_2Cl_2 evolves some heat with stearic acid and with glycerin, and, what is still a greater objection, the reaction is not instantaneous, but takes considerable time for its completion. With drying-oils more heat is certainly evolved than with non-drying ones, but there appears to be no definite relation.

The action of bromine upon unsaturated fatty bodies is instantaneous, and is attended with considerable evolution of heat. It is complete and quantitative, as was lately shown by one of us (*ANALYST*, xx., p. 49). It is not complicated to any extent by secondary reactions; the amount of hydrobromic acid formed measures the substitution, and is very small in most cases. The measurement of the heat evolved promised to supply much more definite data than in the case of H_2SO_4 and S_2Cl_2 .

As the action of bromine upon some of the oils is very violent, it was found necessary to moderate it by the introduction of a diluent, such as chloroform or glacial acetic acid. Owing to its higher boiling-point, the latter has the advantage of allowing of a wider range of rise in temperature, but necessitates the use of the free fatty acids, which without great precaution readily become oxidized. We have satisfied ourselves that there is no heat evolved with either of these bodies. Originally we carried out the reaction in an ordinary test-tube packed with cotton-wool into a beaker, using one gramme of the oil or fatty acid dissolved in 10 c.c. of chloroform and 1 c.c. of bromine. In our later experiments, the results of which we give below, we made use of a vacuum-jacketed test-tube such as is used by Professor Dewar in his experiments on liquid air. From such tubes there is practically no loss of heat through the glass, the only way of escape being from the surface exposed to the outer air. This is, however, a matter of no moment, as the reaction is, as before mentioned, an instantaneous one. As compared with the rise of temperature obtained by the use of an ordinary test-tube, the vacuum-jacketed tube gives results about two degrees higher under the conditions mentioned above as to quantity of substance and solvent used. We believe that the vacuum-jacketed tube might be usefully employed in other calorimetric estimations. Of course, it would be possible to calculate, by making allowance for the heat-capacity of the glass, and the specific heats of the solvent, oil, bromine, and bromination products, the exact amount of heat evolved; but for analytical purposes the observed rise of temperature is fully sufficient. The thermometer used was a standard thermometer divided into fifths of a degree centigrade. As the bromine must be exactly measured, a 1 c.c. pipette was used, connected at the upper end with a narrow tube filled with caustic lime, and having an asbestos plug at each end. The bromine, oil, and solvent were all brought to the same initial temperature.

The following are some of the figures obtained, the calculated iodine number being the rise in temperature multiplied by 5.5.

	Oil or Fat.	Rise of Temperature with Bromine.	Hübl Figure.	Calculated I. No.
1.	Lard	10.6	57.15	58.3
2.	"	10.4	57.13	57.2
3.	"	11.2	63.11	61.6
4.	"	11.2	61.49	61.6
5.	"	11.8	64.69	64.9
6.	"	11.8	63.96	64.9
7.	"	10.2	57.15	56.1
8.	"	10.4	57.8	57.2
9.	"	9.0	50.38	49.5
10.	"	11.0	58.84	60.5
11.	Lard + 10% Cotton-Oil ...	11.6	64.13	63.8
12.	Lard Fatty Acids ...	10.4	59.6	57.2
13.	"	11.0	59.15	60.5
14.	Mutton Fat (Kidney) ...	8.1	44.48	44.5
15.	" " (Flare) ...	7.6	39.7	41.8
16.	Butter	6.6	37.07	36.3
17.	"	7.0	38.60	38.5
18.	" (Fatty Acids) ...	6.2	36.5	34.1
19.	Almond-Oil	17.6	96.64	96.68
20.	Olive-Oil	15.0	80.76	82.5
21.	Maize-Oil	21.5	122	118.2
22.	Cotton-Oil	19.4	107.13	106.7
23.	Castor-Oil	15.0	83.77	82.5
24.	Linseed-Oil	30.4	160.7	167.2
25.	"	31.3	154.9	172
26.	Rape-Oil	18.4	88.33	101.2
27.	"	17.6	77.2	96.8
28.	Cod-liver-Oil	28	144.03	140
29.	Oil sent as Olive-Oil ...	19	108.5	104.5
30.	" " " ...	19.2	105.7	105.6
31.	" " " ...	18.9	105.7	103.9

It will readily be seen from an examination of the above figures that the factor 5.5 expresses the relation not only of one particular kind of fat, but applies also to most kinds of oils which we have examined, whether the iodine absorption be low or high. Thus, a butter having a Hübl number of 38.6 gives an iodine number, calculated from the temperature rise, of 38.5, whilst almond-oil, with 96.64 Hübl number, gives a calculated number of 96.8. Cotton-seed, with 107.13 Hübl number, shows 106.7 calculated, and cod liver, with 144 Hübl number, calculates to 140.

With the two samples of linseed-oil examined the approximation is not good. Now, since linolic acid appears for each molecule of added bromine to evolve as much heat as does oleic acid, as shown by the figures given by cotton-seed and almond-oil respectively, it is probable, to say the least, that the same holds good for linolenic acid. The difference observed in the case of linseed-oil might, on this assumption, be due to one or both of two causes: either the Hübl number does not fully measure, in the case of highly-drying oils, the unsaturated valency of the molecule, or the samples of linseed-oil tested had undergone more or less oxidation, the oxygen or hydrogen group being replaced by the bromine.

As to the former alternative, it is well known that with highly-drying oils, after three hours' action of the Hübl solution even in considerable excess, the maximum of absorption has by no means been reached, and the Hübl number is, therefore, almost certainly too small in these cases. As to the latter alternative, it has been shown by Ballantyne (*Journ. Soc. Chem. Ind.*, 1891, p. 32) that oils, after having undergone oxidation by exposure to air, show a higher Maumené figure than before. We are inclined to think that our calculated number expresses more accurately the real iodine-combining capacity than does the Hübl figure in these cases.

The two samples of rape-oil examined by us do not show any agreement between the observed and calculated iodine number. We believe the samples to be pure, but the Hübl numbers—viz., 88 and 77—are materially lower than the numbers usually accepted for genuine rape-oil. The calculated numbers, on the other hand, obtained by multiplying the rise in temperature by 5.5 agree very well with the normal numbers of genuine rape-oil. It appears very probable, therefore, that the samples of rape-oil examined had undergone a considerable amount of oxidation, which lowered the Hübl number, but did not affect the bromine absorption; that, in fact, the figure calculated from the heat evolution in this, as in the case of linseed-oil, is the correct iodine-absorption number.

We believe that we have established the fact that the heat of bromination, measured as described, affords at the same time the information sought by the Hübl method and that crudely given by the Maumené process. As the whole operation only occupies a minute or so, we think that the method will be found useful in analytical work.

We do not give the factor 5.5 as something absolute. It applies to the particular vacuum-tube used by us and to our precise mode of operation. Each chemist using the method should, by operating upon a sample or two of a non-drying oil with an accurately-determined Hübl number, ascertain the factor for himself.

The conclusions at which we arrive in the foregoing paper may thus be briefly summarized:

1. The Maumené figure stands in some rough relation to the Hübl number, but no definite numerical proportion exists applicable to various oils, as shown by the numbers obtained by various observers.

2. No definite relation appears to exist between the heat evolved by the action of sulphur chloride upon oils and the Hübl number.

3. The rise of temperature observed when bromine acts upon oils stands in proportion to the non-saturation. From such rise the Hübl number can be calculated with very close approach to the numbers obtained by the use of Hübl solution.

4. The accuracy is greater in the case of unoxidized edible fats and oils than in highly-drying oils.

5. The reaction is practically instantaneous.

6. The new method gives at once the Hübl number and what is intended to be given by the Maumené process.

DISCUSSION.

Mr. RICHMOND said he gathered from the authors' remarks upon the Maumené test in the earlier part of the paper that they considered it to be of much less value

than the bromine reaction. He could not agree with the authors in this; in fact, he thought that the two tests were not at all comparable quantitatively. He had been studying the Maumené test for some years, and had come to the conclusion that while a certain part of the rise in temperature might be assigned to the action of the sulphuric acid on the fat, *i.e.*, the hydrolysis of glycerides of the fatty acids, there was a further amount due to the action of the sulphuric acid on the fatty acids, and this latter differed with the various series. Experiments which he had made showed that the rise in temperature resulting from the Maumené reaction varied according to the series to which the fatty acids belonged, and he thought it might be possible to make use of this for estimating the relative proportions of fatty acids of different series present in a sample. In fact, the authors considered the bromine reaction of value, because it gave results equivalent to the iodine absorption, while they condemned the Maumené method because there was no definite relation between it and the iodine absorption. It was on this very ground that he maintained the usefulness of the Maumené reaction as an analytical method; the very fact of its having a different ratio to the iodine absorption in one oil to that in another yielded valuable information not given by the bromine test alone.

Apart from this point, on which he could not agree with the authors, their paper seemed to him of the utmost value, not only because they had devised a rapid and accurate analytical test, but because of the applications of thermo-chemistry to analysis, and their successful attempt, by determining the energy of the reaction, to show the exact nature of the change which was taking place.

Mr. HEHNER said he thought they (the authors) had made it perfectly plain that no attack whatever on the Maumené test had been intended. At the same time it could not be said that the Maumené test had ever been got to give comparable results in the hands of separate observers (although it had been used by many chemists), from causes which one could guess at, but which had never been worked out. The reaction was exceedingly complicated, and the difficulties of working the test were greatly enhanced by the occurrence of side reactions, and by the evolution of sulphur dioxide. On the other hand, the simplicity of the bromine reaction was evident, while its results were obtained in a very short time, and its applicability was increased by the fact that, like the Maumené process, instead of being a volumetric process, it was a simple thermometric method of observation. There was apparently no difference in the amount of heat evolved whether the bromine added were one, two or three molecules, and one might fairly infer from this that the Maumené reaction was also in principle a proportionate one, although Mr. Richmond differed from him in this view. He thought that the method might, in many cases, supply the place of both the Maumené and the Hübl tests, seeing that the indications given by both these tests could be obtained in about one minute with accuracy, but at the same time, as Mr. Mitchell had been careful to point out in reading the paper, it must be taken with a certain amount of caution. With regard to the drying oils in particular, there was something anomalous in the results they had obtained, which pointed to the necessity for further investigation. To edible oils the new method seemed perfectly suited.

Mr. Bevan read the following paper :

THE USE OF FORMALIN AS A PRESERVATIVE OF MILK SAMPLES.

By E. J. BEVAN.

THE commercial article "formalin," which, as is now well known, is a 40 per cent. solution of formaldehyde, has been for some time in use as a preservative for milk intended for sale, and at the last meeting Dr. Rideal read a paper on a method of detecting its presence.

Many analysts, including myself, have for months past been in the habit of using it for the purpose of preserving adulterated samples of milk in case of future reference. Of its value as a preservative agent there can be no doubt, and I would take this opportunity of urging upon public analysts the importance of thus preserving their samples, so as to be in a position, in case of disputed analyses, to refer them to a brother analyst.

My own practice is to add 4 drops of the commercial article to the residue of the sample, which is usually about 4 ounces. As a rule, my samples keep perfectly for six weeks, or even longer. Much depends, of course, on the condition of the milk when the sample is taken. If any considerable amount of decomposition has commenced, the formalin does not entirely prevent the decomposition continuing, but it merely retards it.

The amount of decomposition and consequent loss of total solids is, as a rule, very small, sometimes, in fact, inappreciable; and, as I shall show, in certain cases by no means rare, there is actually an increase in the amount of total solids, especially if the sample be again analysed within a week or two of the first examination. The reason for this I will discuss later.

Some time ago, it occurred to me, that if in the ordinary course of analysis the milk were evaporated with a few drops of formalin, the residue of total solids would be obtained with the minimum of decomposition. As a matter of fact, I found that an almost white residue was obtained, but that the amount was considerably increased. By evaporating the milk with increased quantities of formalin, I found that the total solids were correspondingly increased, as shown in the following table :

TABLE I. :

					Total Solids Per Cent.
Original milk	13.24
5 c.c. evaporated with 2 drops	13.47
" " " 6 "	13.77
" " " 10 "	14.10

Having looked upon formalin as readily volatile, I was somewhat surprised at the result. I then tried experiments, evaporating various substances with formalin. The results are shown in the following table :

TABLE II. :

<i>Action on a Mixture in equal parts of Albumen and Milk-Sugar.</i>				Total Solids Per Cent.
5 c.c. evaporated alone	7.59
" " with 1 c.c. formalin	9.29
<i>Action on Albumen.</i>				
5 c.c. evaporated alone	3.82
" " with 1 c.c. formalin	4.12
<i>Action on Milk-Sugar.</i>				
5 c.c. evaporated alone	4.62
" " with 1 c.c. formalin	7.28
<i>Action on Cane-Sugar.</i>				
5 c.c. evaporated alone	4.82
" " with 1 c.c. formalin	6.90

It appeared probable, from a consideration of these numbers, that the formalin had in every instance entered into combination. Part of the effect produced might of course have been produced by polymerization of the aldehyde and conversion into a non-volatile body. I therefore evaporated 5 c.c. of water and 1 c.c. of formalin, but got no residue. On evaporating salt solution with formalin I got a decided increase of weight, and there was left behind a small quantity of a white substance insoluble in water. From this it appears probable that a small part at least of the increase of weight observed is due to the formation of this non-volatile polymer.

Then, again, part of the increase may be due in the cases of milk and cane sugar to the conversion into galactose and dextrose. The gummy appearance of the residue left on evaporation with formalin suggests this as probable, but owing to press of work I have been unable to prove it.

We have hitherto been considering the effect due to relatively considerable quantities of formalin; we will now discuss the phenomenon of the increase of total solids in milk which has been preserved with small quantities of formalin.

Some time ago, I examined a sample of milk which gave, as the mean of two very closely-agreeing numbers, 11.60 per cent. of total solids. On hearing that the sample was referred to Somerset House, I analysed it again, and found that the total solids had increased to 11.71. I sent portions of my sample to Messrs. Chattaway, Dyer, and Hehner, and in each case their results were higher than the original 11.60 per cent.

This result appeared so extraordinary, having in view the fact that I had only added 4 drops of formalin to 4 ounces of the milk, that I made some further experiments. I took a sample of fresh milk, which gave 12.145 per cent. of total solids. To it I added formalin in the proportion of 1 drop to the ounce, and I found that at the end of seven days the total solids had increased to 12.286 per cent., and in fourteen days they amounted to 12.21 per cent. I could quote several other cases in my own experience in which the total solids had similarly increased. I will content myself with recording two cases, kindly furnished by Mr. E. W. Voelcker, which amply confirm my own results. In one case, using 4 drops to 6 ounces, he found

that in fourteen days the total solids had risen from 12.48 to 12.64, and in another from 11.62 to 11.82. Assuming that the whole of the formalin had simply been retained by the milk, the increase would only account for about one-fourth of the actual increase observed. It is therefore necessary to account for it in some other way. In all probability it is largely due to the conversion of milk-sugar into galactose. Assuming that there is 4 per cent. of milk-sugar, and that the whole of it is converted into galactose, this would mean an increase in the total solids of 0.2 per cent. In the cases I have recorded the increase amounts to 0.14 and 0.11 per cent., and in Mr. Voelcker's samples the increase was 0.16 and 0.20. I regret that I have been unable to pursue the matter further, but trust that the facts I have brought to the notice of the Society may not be without interest.

Mr. Boseley read the following paper :

NOTE ON THE DETECTION OF FORMALIN.

By H. DROOP RICHMOND AND L. KIDGELL BOSELEY.

A SOLUTION of formaldehyde, called "formalin," having come into use as a food preservative, it becomes of importance to be able to detect and estimate it.

The literature of formaldehyde is very voluminous, and numerous tests for it have been proposed. As is well known, aldehydes reduce Fehling's solution and ammoniacal silver nitrate, and give Schiff's reaction. These reactions, however, are by no means characteristic of the aldehyde.

Legler's method for the estimation of formaldehyde (*Ber.*, xvi., 1333) by titration with ammonia is to a certain extent characteristic of formaldehyde, but is not applicable to dilute solutions. Legler states that 3 molecules of ammonia are equal to 4 of formaldehyde, while Lösekan (*ibid.*, 22, 1565) maintains that 3 molecules are equal to 6 of formaldehyde. This discrepancy is explained by Eschweiler (*ibid.*, 22, 1929), who shows that with methyl-orange, cochineal, tropæolin, and congo-red 6 molecules are indicated, while with litmus and phenolphthalein only 4. This is due to the acid reaction of the hexa-methylene-tetramine formed.

Plöchl (*ibid.*, 21, 2117) states that when a neutral solution of formaldehyde is mixed with ammonium chloride it becomes acid; on heating CO_2 is evolved, and trimethylamine is formed.

Kleeberg (*Annalen*, 263, 283) shows that formaldehyde combines with phenols in the presence of HCl , but he did not succeed in purifying the compounds formed.

Pulvermacher, in a series of papers, describes many condensation products with substituted ammonias; and also shows (*Ber.*, 26, 2360) that the very insoluble formalazine is produced by mixing formaldehyde and hydrazine hydrate; this yields a platino-chloride $(\text{C}_2\text{H}_4\text{N}_2)_6\text{H}_2\text{PtCl}_6$.

Trillat (*Compt. Rend.*, 116, 891) gives the following tests for formaldehyde: The solution is to be mixed with dimethylaniline acidified with sulphuric acid and agitated. After heating for thirty minutes on the water-bath, it is made alkaline, boiled until the smell of dimethylaniline has disappeared, then filtered. If the filter-paper be

moistened with acetic acid, and powdered lead oxide be sprinkled on it, a blue colour, due to the formation of tetra-methyl-diamido-diphenylmethane is produced if formaldehyde is present. Or the formaldehyde solution may be mixed with a solution of aniline (3 grammes to 1 litre), when a white precipitate of anhydro-formaldehyde aniline appears, which may be weighed. A precipitate is also given by acetaldehyde. Trillat says that, as formaldehyde easily forms condensation products, it is not always detected in food after a lapse of time.

Three years ago one of us worked with formaldehyde as a preservative for milk, and used as a method for its detection the reduction of Fehling's solution, or of ammoniacal silver nitrate solution. Quite recently Thomson (*Chem. News*, lxxi., 247) has proposed the use of the latter, and, although he modifies the test by working in the cold, he does not succeed in obtaining a reaction which is characteristic of formaldehyde.

Schiff's reagent has been used as a test for formaldehyde. It is, however, very unsatisfactory unless care be taken, for if an excess of sulphurous acid is used, no reaction is obtained with traces of formaldehyde, and any alkali combined with an acid weaker than SO_2 also gives a red coloration. The red coloration appears on warming Schiff's reagent, on blowing air through it, or even on placing it in an uncorked bottle, so that unless great precautions are taken the test is unreliable. Still, it is useful as a confirmatory test. In applying it as such to milk, we precipitate the casein with a little sulphuric acid, filter, and then add a little Schiff's reagent to the filtrate; any red colour which may appear roughly indicates the amount of formaldehyde present.

Another test which we believe to be well known, though it has not actually appeared in print, was pointed out by Mr. Hehner. It is the formation of a blue colour when milk, formaldehyde, and sulphuric acid are mixed together. This was first brought to our notice by Mr. Bevan, who had obtained a blue colour in a Leffmann-Beam experiment, which he could not account for. We suggested that it might be due to formaldehyde, but we were unable to obtain the reaction with other milks, owing to our having used an excess of formaldehyde. We find that when formaldehyde is in large quantity, say 0.5 per cent., no blue colour is obtainable.

We have since found that the above reaction is due to the albuminoids of milk. We have also obtained it from egg albumen and peptone, but not from gelatin. To obtain the reaction it is only necessary to add sulphuric acid (94 per cent. H_2SO_4 gives the best results) to the milk, when a blue ring is formed at the junction of the two fluids. The food suspected may be distilled and the formaldehyde obtained in plain aqueous solution; but we prefer the use of peptone for testing, as the blue colour is not then obscured by the charring of the organic matter by the acid.

Bearing in mind Pulvermacher's researches, we have found a reaction between formalin and diphenylamine. A solution of diphenylamine in water is made, just sufficient sulphuric acid being added as will effect solution. The liquid to be tested (or the distillate) is added to this solution and boiled. In the presence of formaldehyde a white flocculent precipitate is deposited, which is often coloured green if the acid used contained nitrates.

We find it most convenient to distil into the diphenylamine solution and then boil. This simple test we believe to be characteristic of formaldehyde.

We are engaged in determining the composition of the precipitate, and in working out the quantitative estimation of formaldehyde in this manner.

We are able to confirm Trillat's observation that after a certain time formaldehyde cannot be detected. We can obtain the reaction in milk which has not curdled.

We think from the list of methods enumerated that there is not the slightest difficulty in definitely proving the presence of formaldehyde in foods when present. Hehner's reaction, confirmed by the diphenylamine test, Schiff's test, and those proposed by Trillat, Pulvermacher and Plöchl, should be amply sufficient.

DISCUSSION.

Mr. M. A. ADAMS said he could confirm Mr. Bevan's observations as to the preservative power of formalin, having used it successfully for several years. His practice was to add it immediately on receipt of the sample before the analysis was started; four drops to a third of a pint was quite sufficient to preserve samples for a length of time.

He had found that the growth of moulds, unlike bacterial growth, was not inhibited by formalin.

Mr. RICHMOND remarked that he noticed that Mr. Bevan had found it necessary to add the formalin while the milk was still fresh, and if it was allowed to turn, formalin failed to keep it without decomposition; this fact he had also observed in connection with the use of hydrofluoric acid as a preservative some years ago. With regard to the increase in the total solids, an immense number of compounds had been prepared from formaldehyde by condensation in the presence of various bodies, and he thought it not improbable that such an action might occur when formalin was added to milk, the resulting compounds accounting for some, at any rate, of the increase. It had been found, for instance, that formaldehyde condensed very easily in the presence of lime, giving formose, and in view of the alkaline salts present in milk, it was not difficult to imagine the formaldehyde becoming converted into formose, or some other compound, the weight of which would be added to that of the total solids.

Mr. W. W. FISHER said he had made the experiment of boiling milk containing formalin with hydrochloric acid, and had obtained a blue reaction, developing into purple, the colour disappearing on more prolonged boiling. He had not worked it out, but thought that if it could be really established it might prove useful as a test.

Mr. C. A. SEYLER said that he had obtained in using Schmid's process a yellow colour, when formalin was present. In its absence he had noticed a pale violet colour at first, soon masked, however, by the yellow of caramel. On dissolving cheese in hydrochloric acid a distinct pale violet was produced, but in the presence of formaldehyde the casein was coloured a strong yellow, and became much less soluble. There was no doubt that formaldehyde had a considerable action on albuminoids. Albumin seemed to be rendered insoluble (although not precipitated at once), and, when once precipitated, it could not be got back into solution again. He had found that milk to which formalin had been added after curdling could not be brought to a thin liquid state by shaking up with ammonia.

Dr. DYER inquired whether formalin had any influence on the proportion of fat as determined by any method, or whether it was simply the non-fatty solids that were affected.

Mr. BEVAN said he had not made any direct experiments on the action of formalin on fat; all he could say was that the total percentage of fat in the milk was not affected, as far as was indicated by ordinary methods of estimation.

He could exactly confirm what Mr. Seyler had said as to the casein being rendered insoluble after precipitation. The action of formalin on gelatin was tolerably well known, and numerous patents had been taken out in connection with it for the purpose of rendering gelatin insoluble.

Like Mr. Adams, he had sometimes found moulds on the top of milk samples that had been kept for some time.

Mr. BODMER said he wished to again raise the question as to whether it would not be well, in face of the growing use of formalin in the trade, that some understanding should be come to by the Society as to the action of its members in the event of their detecting the presence of formalin in samples passing through their hands officially.

Dr. SYKES called attention to the recent investigations of Weigle and Merkel, who had found that the addition of formalin to milk rendered the casein indigestible.*

The CHAIRMAN (Mr. Otto Hehner) said that although he was fully aware of the importance of the point (especially as formalin was being used for other articles besides milk), the subject which it opened was so very wide that the Society could not deal with it on the present occasion, and he therefore hoped that Mr. Bodmer would not press his suggestion.

ON FORMALIN AS A MILK-PRESERVATIVE.

BY SAMUEL RIDEAL, D.Sc.

(Read at the Meeting, May 1, 1895.)

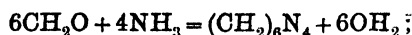
In my own experiments with formalin as a milk-preservative, I have found that when used in the proportion of 1 part formaldehyde in 10,000, milk remained fresh without curdling for seven days. Mixtures of water and milk containing formaldehyde in the proportion 1 : 10,000 and 1 : 100,000 were also tested. The control sample became sour on the third day, that with formaldehyde 1 : 100,000 remained sweet for one day longer, and that with 1 : 10,000 was fresh on the twelfth day. I found that the formalin used for preserving milk in the trade has 5 oz. of pure formalin to 1 gallon, corresponding to 2 oz. formaldehyde in 160 oz., or 1 : 320. This is used in the proportion of $\frac{1}{2}$ pint to the churn of 17 to 18 gallons, and does not impart any taste or smell to the milk even after boiling. In this strength the milk keeps fresh for at least three days, and corresponds to :

1 part formaldehyde in 46,080 parts milk,
or, 1 c.c. formalin in 18,432 c.c. milk.

* See abstract on page 167.

One gallon of the diluted formalin as used by the milk-vendors does the same work as 10 lb. of the powder preservative containing 75 per cent. of boric acid and 25 per cent. of borax.

I understand that 15,000 gallons of this dilute formalin have been sold to milk-vendors since it was placed on the market last year. At present I have not been able to devise a simple method for estimating the amount in milk. In strong solutions it can be estimated by means of a standard ammonia solution, since, as was shown by Trillat, formaldehyde combines with ammonia to form hexamethylene amine, thus :



so that 180 parts by weight of formaldehyde combine with 68 of ammonia.

In determining the strength of a formalin solution, first ascertain its acidity (this I have never found to be higher than 6 c.c. of $\frac{\text{N}}{100}$ NaHO per 100 c.c. of pure formalin), agitate a known volume with excess of a standard ammonia solution in a stoppered bottle, and then distil off the excess of ammonia into standard acid and titrate back.

For detecting formaldehyde in milk, I find Schiff's reagent (magenta bleached by sulphurous acid) of use. All milk samples which I have examined give, however, a pink colour with this reagent, pointing to the presence of some aldehydic compound in milk, but it differs from formaldehyde in not being volatile. If, therefore, a portion of the suspected milk sample be distilled into water, the distillate gives a pink colour with Schiff's reagent if formalin is the preservative employed.

I consider that formalin is much to be preferred to borax or boric acid as a milk-preservative, seeing that the quantity required is so much smaller, viz., 1 oz. of formaldehyde, as against 5 lb. of borax and boric acid. Its volatility is distinctly in its favour, as the small quantity present is evaporated on warming the milk. As to its toxic action, I have not heard of any ill effects, and have myself repeatedly drunk the 1 per cent. solution, whilst that used for milk preservation is almost tasteless. For cream a slightly stronger solution is used, and in this connection it may be interesting to note that the use of salicylic acid in conjunction with the borax-powder has increased in the last year or two, being present to the extent of 5 to 10 per cent. in some preservatives, mixed with saccharin to mask the taste.

DISCUSSION.

Mr. WYNTER BLYTH raised the question as to whether formalin should not be considered an adulterant, and suggested that steps might with advantage be taken by the Council of the Society to get the matter put upon a statutory basis. It was very desirable that a clear definition should exist as to the nature of a preservative, and the proportions in which such might be used.

Mr. CASSAL agreed with Mr. Wynter Blyth. Public Analysts were in a very difficult position owing to the want of a clear understanding as to what should be done about preservatives. The Local Government Board had been approached on the matter in regard more especially to the addition of boric acid preparations to butter, and had expressed a very guarded opinion. The Board did not appear to consider it advisable that boric acid in butter should be regarded as an adulterant,

at any rate at present. Public authorities and the public generally appeared to take a different view. His own opinion was, that to permit the addition of preservatives to food was objectionable and, in fact, dangerous. If preservatives were to be allowed, purchasers should be fully informed at the time of purchase of the nature and quantity of the preservative used.

The CHAIRMAN (Mr. Allen) said it was open to question whether the Council would be able to take any effective action; the practical difficulties in the way were considerable. He was afraid it would not be in order to submit Mr. Blyth's suggestion to the meeting as a formal resolution, since no previous notice had been given.

Mr. BODMER remarked that the Sale of Food and Drugs Act appeared to sanction the use of preservatives, provided they were not injurious to health.

Dr. RIDEAL said it was pretty certain that formalin was a preservative which was not injurious to health. The fact of so small a quantity of it being required was very much in its favour. He did not know what was the amount of a toxic dose of formaldehyde, but he had himself repeatedly drunk 1 per cent. solutions of it without ill effects.

A COMPARISON OF THE ORGANIC CARBON AND NITROGEN RESULTS OBTAINED BY DR. FRANKLAND AND THE COMPANIES' ANALYSTS FROM THE WATERS SUPPLIED BY THE METROPOLITAN WATER COMPANIES.

By W. C. YOUNG, F.I.C., F.C.S.

(Read at the Meeting, May 1, 1895.)

ONE of the most important duties a public analyst is called upon to discharge is to advise a sanitary authority as to the quality of the public water-supply, especially with reference to pollution by organic matter. In the districts supplied by the London Water Companies it occasionally happens that the public analyst's opinion as to the quality of the water is opposed to that of Dr. Frankland, who reports monthly to the Local Government Board. This arises from the fact that on the one hand Wanklyn's "ammonia" process is used, and, on the other, Frankland's "combustion" process is employed. It seems late in the day to raise the question of the reliability of Frankland and Armstrong's combustion process, but as the matter is of great public importance, and has, so far as I know,* never been publicly discussed since the historical dispute between its authors and Mr. Wanklyn, I venture to bring to your notice a comparison of the results obtained by it in the analyses of the London Companies' water made by Dr. Frankland on behalf of the Local Government Board and by the chemists employed by the Water Companies. These analyses are published in the monthly reports of the Official Water Examiner to the Local Government Board, from which documents I have taken my data.

Dr. Frankland claims for his process that it accurately determines the quantity of carbon and nitrogen contained in the organic matter present, and, further, that the ratio of carbon to nitrogen indicates its origin.

If the process gave accurate results, Dr. Frankland's figures should differ little

* This paper was written in June, 1894.

from those of the companies' chemists, and although the samples taken in the same month of any one of the companies' water, analysed by either, may possibly have contained a little more or less organic matter, the organic matter must have had a fairly constant composition; therefore the ratio of carbon to nitrogen should be practically the same in each case.

In the table on the opposite page I have placed side by side the ratio of carbon to nitrogen (nitrogen = 1) shown in the results obtained by the companies' analysts and by Dr. Frankland during the three years, 1891, 1892, and 1893.

I should mention that Dr. Frankland's analyses include only one sample of each company's water per month, but the companies' analysts examine several. I have taken in every case the mean of the latter, but much greater discrepancies are shown by particular samples.

It will be seen by this table that, with very few exceptions, Dr. Frankland's results differ greatly from the others, and, except in the case of the Kent Company, show a much higher ratio of carbon to nitrogen, and, further, that the ratios are much less uniform.

The ratios of carbon to one of nitrogen in the two sets of results vary as follow :

1891.			Companies' Analysts			Dr. Frankland.		
New River	2.9	to	4.4	...	3.6	to 8.1
East London	3.3	„	4.3	...	5.2	„ 9.6
Chelsea	3.3	„	5.5	...	3.9	„ 10.5
West Middlesex	3.3	„	5.3	...	3.6	„ 10.0
Lambeth	3.5	„	6.7	...	4.1	„ 9.8
Grand Junction	3.2	„	5.6	...	3.8	„ 7.9
Southwark	3.6	„	6.2	...	3.4	„ 9.5
Kent	3.8	„	22.1	...	3.4	„ 6.6
1892.								
New River	3.0	„	4.8	...	4.1	„ 11.2
East London	3.1	„	5.0	...	4.9	„ 9.8
Chelsea	3.6	„	4.8	...	6.2	„ 10.5
West Middlesex	2.6	„	5.3	...	6.3	„ 10.7
Lambeth	3.2	„	4.9	...	5.5	„ 10.4
Grand Junction	3.1	„	5.6	...	6.1	„ 9.4
Southwark	3.2	„	5.2	...	5.4	„ 10.5
1893.								
New River	2.1	„	4.5	...	3.8	„ 8.3
East London	3.1	„	4.0	...	2.1	„ 8.1
Chelsea	2.3	„	4.0	...	4.5	„ 10.3
West Middlesex	2.4	„	4.7	...	4.8	„ 9.3
Lambeth	2.6	„	4.8	...	6.0	„ 9.6
Grand Junction	2.9	„	4.9	...	3.6	„ 9.0
Southwark	2.7	„	4.8	...	3.9	„ 11.4

According to these results the Companies' chemists (except the Kent Company's) find that the composition of the organic matter in the water nearly approaches that of animal matter, while Dr. Frankland's rather indicate that it is generally of vegetable origin. In fact, the two sets of results differ almost completely. The actual analytical results show many remarkable and striking discrepancies. In the great

TAB. SHOWING RATIO OF CARBON TO NITROGEN (NITROGEN = 1) IN THE RESULTS OF ANALYSES OF THE WATER SUPPLIED TO LONDON BY THE METROPOLITAN WATER COMPANIES

MADE BY DR. FRANK AND THE WATER COMPANIES' ANALYSTS.

1891.

Name of Water Company.	JANU. ARY.		FEBRU. ARY.		MARCH.		APRIL.		MAY.		JUNE.		JULY.		AUGUST.		SEPT.-BER.		OCTOBER.		NOVEM.-BER.		DECEM.-BER.	
	Company's Analysts.	Frankland.	Company's Analysts.	Frankland.	Company's Analysts.	Frankland.	Company's Analysts.	Frankland.	Company's Analysts.	Frankland.	Company's Analysts.	Frankland.	Company's Analysts.	Frankland.	Company's Analysts.	Frankland.	Company's Analysts.	Frankland.	Company's Analysts.	Frankland.	Company's Analysts.	Frankland.	Company's Analysts.	Frankland.
New River	3.8	3.6	2.9	8.1	3.4	7.	4.	5.1	3.8	6.7	3.3	6.8	4.	4.6	4.	5.3	3.7	5.	3.9	5.4	4.4	4.6	3.8	5.2
East London	4.	5.5	4.2	6.6	4.1	5.6	3.3	6.9	3.9	7.7	4.	7.8	3.6	6.	3.7	5.2	4.1	7.	3.4	6.9	4.	9.6	4.3	6.3
Chelsea	4.	6.1	4.6	7.3	3.6	4.4	4.4	8.4	3.6	10.5	4.	6.3	3.3	5.6	5.5	5.4	3.7	6.3	3.5	6.3	3.8	6.8	5.5	3.9
West Middlesex.	3.5	3.6	6.3	8.3	3.7	3.7	3.7	3.6	7.8	3.6	7.3	3.6	7.3	5.7	3.6	5.5	4.3	7.5	4.3	8.5	4.8	10.	4.7	6.4
Lambeth	6.7	4.1	4.4	9.5	3.5	6.7	4.6	4.1	4.2	8.5	3.8	6.0	3.6	7.	3.6	6.9	4.5	7.4	4.7	9.8	4.7	8.2	4.4	6.4
Grand Junction	3.4	3.8	5.	7.3	3.5	6.9	3.8	6.6	3.5	7.6	3.4	5.4	3.5	6.	4.	6.3	4.	7.9	3.2	7.1	5.6	6.3	4.9	7.2
Southwark	4.1	3.4	5.8	7.9	4.6	9.5	4.	6.2	4.6	7.9	4.7	3.9	3.6	6.	4.7	4.2	4.	7.3	6.2	8.7	5.3	6.6	5.	6.2
Kent	5.6	4.7	12.9	4.4	16.2	6.6	3.2	5.5	4.2	4.6	10.	5.2	13.5	5.2	3.8	4.9	6.2	3.5	6.8	3.4	22.0	4.7	22.1	3.4
1892.																								
New River	3.9	7.	3.	5.	4.3	5.5	3.5	5.1	3.5	4.1	3.9	4.8	3.7	5.9	4.	5.1	4.3	6.9	3.5	11.2	4.8	9.	3.8	4.5
East London	5.	6.9	3.3	5.4	3.7	6.8	3.9	5.9	4.2	4.9	3.9	5.9	3.1	8.8	3.5	6.7	3.7	5.5	4.1	8.1	4.5	9.8	3.9	5.5
Chelsea	4.8	7.4	4.6	7.1	4.6	9.	3.7	7.9	4.2	8.	3.8	6.3	3.6	6.2	3.9	9.7	4.8	6.8	4.2	10.5	4.6	10.5	3.6	10.2
West Middlesex.	3.5	8.	5.3	7.1	4.6	9.2	4.5	6.3	4.9	7.5	2.6	6.3	3.6	6.5	3.1	9.	3.4	10.7	3.3	9.9	5.3	8.	4.5	7.3
Lambeth	3.7	9.7	4.9	5.8	4.2	8.8	4.4	5.5	4.2	7.5	4.1	6.5	3.7	5.6	3.2	6.7	3.3	9.9	4.	10.4	4.9	9.	4.2	9.3
Grand Junction	3.4	6.1	4.7	6.7	3.9	9.4	4.3	7.	4.2	8.2	3.4	6.4	3.4	8.	3.3	7.8	3.1	7.4	3.7	7.9	5.6	6.5	4.6	7.8
Southwark	3.4	6.3	5.2	5.4	4.8	8.3	4.1	7.	4.2	6.3	3.9	6.	4.1	5.4	3.7	7.4	3.2	10.3	3.9	10.5	4.7	10.2	4.7	6.6
1893.																								
New River	3.	4.3	3.2	4.	4.5	5.3	2.7	7.	3.6	7.6	3.1	7.3	2.7	3.8	2.7	8.3	2.1	6.1	2.8	7.6	2.8	5.	2.6	8.8
East London	3.7	4.9	3.8	8.1	4.	4.5	3.4	7.	3.4	2.1	3.4	8.1	3.2	5.4	3.5	6.4	3.1	7.	3.3	6.5	3.2	6.6	3.5	6.6
Chelsea	4.	5.9	3.7	6.6	3.9	7.1	3.8	4.5	2.4	3.8	3.2	7.	2.3	5.8	2.6	4.5	2.6	5.5	2.5	4.7	2.6	10.3	3.3	8.8
West Middlesex.	4.1	4.8	4.7	7.6	4.7	6.2	3.5	6.	3.4	8.4	3.5	8.7	3.3	7.3	3.4	8.2	2.4	5.2	3.4	8.	2.6	8.5	3.4	9.3
Lambeth	3.8	6.	4.6	6.1	4.6	6.1	3.4	9.	3.3	6.8	3.	7.5	2.9	7.	3.3	7.1	2.6	6.7	3.8	6.8	3.4	7.	4.1	9.6
Grand Junction	4.2	5.	4.9	6.1	3.9	6.1	3.4	5.7	2.9	6.6	3.	7.6	3.1	4.9	3.	3.6	2.9	7.3	3.	7.3	3.4	6.7	3.6	9.
Southwark	4.2	3.9	4.8	4.8	4.2	7.6	3.2	4.8	3.3	5.4	3.1	9.6	3.2	5.8	3.	5.1	2.7	4.4	3.5	5.1	3.3	7.	4.1	11.4

majority of cases the organic nitrogen found by the Companies' chemists is much higher, and the organic carbon often much less, than Dr. Frankland's. A few of the most noticeable are the following:

1891.—As compared with Dr. Frankland's results, the Kent Company's chemist found nearly twice as much nitrogen in January, three times as much in February, about half as much in March, less than half in July, less than one-third in October, one-fourth in November, and less than one-third in December.

The New River Company found twice as much nitrogen in February, more than three times in March, more than twice in May and June, and nearly twice in October.

The East London Company found more than twice as much nitrogen in April, and nearly twice in May and November.

The Chelsea Company's nitrogen was more than twice as much in May.

The West Middlesex Company's nitrogen was more than twice in March, May, and November.

The Lambeth Company's nitrogen was nearly twice in February, May, and November.

The Grand Junction Company's nitrogen was more than twice in May.

The Southwark Company's nitrogen was nearly twice in February and May, and more than twice in March.

1892. *Jan.* The West Middlesex and the Southwark Companies' nitrogens were nearly, and the Lambeth Company's more than, twice.

The New River Company's carbon was one-half.

March. The East London, Chelsea, and Lambeth Companies' nitrogens were nearly, the New River Company's exactly, and the West Middlesex and Grand Junction more than, twice.

April. The East London Company's nitrogen was nearly, and the Chelsea Company's more than, twice.

May. The West Middlesex and Lambeth Companies' nitrogens were nearly, and the Chelsea and Grand Junction Companies' exactly, twice.

June. The West Middlesex and Grand Junction Companies' nitrogens were nearly twice.

July. The West Middlesex Company's nitrogen was nearly, the East London and Grand Junction Companies' more than, twice, and the West Middlesex more than three times.

August. The East London Company's nitrogen was nearly twice, the Grand Junction more than twice, and the West Middlesex more than three times.

Sept. The New River Company's nitrogen was nearly, the West Middlesex, Lambeth, and Southwark Companies' more than, twice.

Oct. The West Middlesex, Lambeth, and Southwark Companies' nitrogens were nearly, and the New River more than, twice.

1893. *Dec.* The Chelsea Company's carbon was less than half.
- Jan.* The Chelsea Company's carbon was less than one half.
- Feb.* The East London Company's carbon was about one half.
- March.* The Lambeth and Grand Junction Companies' carbons were about, and the Southwark exactly, one half.
- April.* The East London Company's nitrogen was nearly, and the New River, Lambeth, and Grand Junction Companies' more than, twice.
- The Chelsea Company's carbon was less than one half.
- May.* The Chelsea, Lambeth, and Grand Junction Companies' nitrogens were nearly, and the New River and West Middlesex Companies' more than, twice.
- The Chelsea Company's carbon was less than one half.
- June.* The Lambeth and Grand Junction Companies' nitrogens were nearly, and the New River, East London, West Middlesex, and Southwark Companies' more than, twice.
- The Chelsea Company's carbon was less than one half.
- July.* The East London Company's nitrogen was nearly, the New River Company's exactly, and the West Middlesex and Lambeth Companies' more than, twice.
- The Chelsea Company's carbon was less than one half.
- August.* The West Middlesex and Lambeth Companies' nitrogens were exactly twice, the East London more than twice, and the New River three times.
- The Chelsea Company's carbon was less than one half.
- Sept.* The West Middlesex Company's nitrogen was twice, the East London and Lambeth Companies' more than twice, and the New River and Grand Junction Companies' nearly three times.
- The Chelsea Company's carbon was less than one half.
- Oct.* The New River and West Middlesex Companies' nitrogens were more than twice.
- Nov.* The New River Company's nitrogen was twice, and the East London, West Middlesex, and Grand Junction Companies' nearly twice.
- The Chelsea Company's carbon was less than one third.
- Dec.* The New River Company's nitrogen was nearly, and the West Middlesex, Lambeth, Grand Junction, and Southwark Companies' more than, twice.

The Chelsea Company's carbon was little more than one half.

As might have been anticipated, the organic nitrogen results show the most frequent discrepancies, but it is curious that the carbon results of the Chelsea Company's analyst should have been so very much less than Dr. Frankland's each month (with a few exceptions), from December, 1892, to December, 1893.

These comparisons show very plainly how extremely unreliable the process is; and if three sets of analyses by eminent chemists (one of them the originator), who

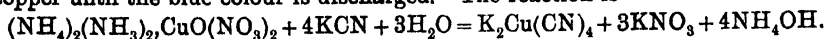
have had almost daily practice with it, differ so enormously, how can an analyst, having only to apply it very occasionally, be expected to place any faith in it?

Since the above was written, the Companies' analysts in their report for July, 1894, explain the discrepancies between Dr. Frankland's results, in the case of the Chelsea Company's water, and their own, by stating that the samples were taken from standpipes, which were found, on inquiry, to be supplied by the New River Company.

They make no reference to the equally serious and more numerous discrepancies in the results of the other Companies' waters, but in their report for August, 1894, they state: "We have recently adopted certain modifications in the estimation of organic nitrogen, suggested by Dr. Frankland. . . . These seem to give more accurate results." I have compared all the results published since, and find that the differences are not so great as formerly, but in many instances they are large enough to completely condemn the process.

The Wet Assay for Copper. R. S. Dulin. (*Journ. Amer. Chem. Soc.*, 1895, xvii, pp. 346-351.)—The methods in general use for rapidly determining copper are the cyanide method, the iodide, and the electrolytic.

In the cyanide method, potassium cyanide is run into an ammoniacal solution of copper until the blue colour is discharged. The reaction is—

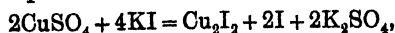


The following precautions should always be observed:

1. The bulk of the liquid to be titrated should always be the same.
2. The solution must be at the temperature of the laboratory.
3. The amount of ammonia added must be nearly constant. Neglect of these precautions may cause errors of several per cent.

Cadmium interferes with the reaction, and to obviate this the author suggests precipitating the copper by boiling with aluminum foil, leaving cadmium in solution, dissolving the precipitated copper in HNO_3 , treating the solution with NH_4OH , and then titrating. Silver also interferes, but in a regular manner, and when the amount is known may be allowed for.

The iodide method depends on the reaction



the amount of iodine being determined by $\text{Na}_2\text{S}_2\text{O}_3$. The precautions to be observed are—

1. The presence of iron in about equal amount with the copper requires more $\text{Na}_2\text{S}_2\text{O}_3$, increasing the amount of copper two or three per cent.
2. The solution should be titrated cold.
3. Large amounts of alkaline salts, especially sodium sulphate, decrease the amount of copper.
4. Bismuth interferes with the end reaction.

With the modification of precipitating the copper first with aluminum, the results are nearly as accurate as with the modified cyanide method, but more time is required.

The chief source of error in the electrolytic method is the deposition of other metals with the copper. The author obviates this by making the deposition from a solution containing a large amount of nitric acid. The best results are obtained with 20 c.c. of HNO_3 to 150 c.c. of solution, but a stronger current than usual is required to precipitate all the copper.

The following are the results obtained on estimating a copper matte containing 20.15 per cent. of copper as determined by many analyses by different assayers, another containing 28 per cent., and an ore containing 30.18 per cent. :

		Various Analyses.			
		Copper.	Cyanide Method.	Iodide Method.	Electrolytic.
Matte	20.15	20.15	20.25	20.045
Matte	28	27.95	28.35	28.15
Ore	30.18	30.20	30.3	30.05

In the cyanide and iodide methods the copper was first precipitated with aluminum foil.

The general conclusion arrived at is that, while the iodide method is usually one-tenth to three-tenths per cent. too high, the electrolytic method is too high or too low according to the amount of metallic substances that can be precipitated by the current. The cyanide method gives results practically correct. C. A. M.

The Separation of Solid and Liquid Fatty Acids. E. Twitchell. (*Journ. Amer. Chem. Soc.*, 1895, xvii., pp. 289-295.)—The most promising processes are based on the greater solubility in ether of the lead soaps of the liquid acids first noticed by Varrentrapp. Having failed to obtain a satisfactory separation, the author has made a study of various methods based on this process.

1. **Muter's Process** (ANALYST, 1889, p. 61).—The following results were obtained with lard fatty acids prepared with the usual precautions.

			Iodine absorbed per cent.	
Lard	56.27
Fatty acids	{ 58.49
				{ 59.26
Liquid acids by above process	94.06

The iodine number of the liquid acids is in agreement with that found by Muter for lard, but the author proves that lead soaps of saturated fatty acids are soluble in ether, 100 c.c. dissolving 0.015 of the lead salts of purified commercial stearic acid (Iodine No. 0) at 0° C.* Another serious objection is the oxidation caused by the exposure of the lead soaps to the air.

2. **A Modification of Jean's Method.** (*Chimie Analytique des Matières Grasses.*)—Four grammes of the same lard fatty acids were dissolved in 50 c.c. of 95 per cent. alcohol and 2.5 grammes of lead acetate in 20 c.c. of the same alcohol added, both solutions being hot. There was an immediate precipitate, which was allowed to stand at the laboratory temperature for one hour, and then at 15° C. for another hour. A part was then filtered into a separating funnel, treated with ether and HCl, the acids washed and dried in a current of CO_2 , their iodine number determined, and their

* Cf. Hahner, ANALYST, xvii., p. 181.

percentage in the original solution calculated. The precipitate was washed with 95 per cent. alcohol, decomposed with HCl, and the solid fatty acids dried and weighed. Their iodine number was also determined. The results obtained were :

	Per cent. obtained.	Iodine No.
Solid fatty acids	46.24	4.9
Liquid fatty acids... ..	51.82	103.37

These figures indicate a fractional precipitation in which all the solid and part of the liquid acids were precipitated. The difference between the iodine number of the liquid acids by this process and by Muter's, shows that in the latter case a considerable amount of saturated or of oxidized fatty acids must have been present.

To determine whether the liquid acids were quite free from solid acids, and also whether the process was really a fractional precipitation, the author made the following experiment : 4 grammes of lard fatty acids were dissolved in 95 per cent. alcohol, precipitated with lead acetate, and the precipitate filtered after an hour. Ten c.c. of the filtrate were drawn off and the fatty acids recovered, while the remainder of the filtrate was kept at 0° C. for an hour, when there was an additional precipitate, which was also filtered off.

The iodine absorbed by the fractions was :

	Amount obtained. Gramme.	Iodine No.
Lard fatty acids	—	62.57
Fatty acids from filtrate at 15° ...	0.02675 (46.81 %)	109.35
„ „ precipitate at 0° ...	0.1020	—
„ „ filtrate at 0° ...	0.1915	118.02

The fatty acid obtained from the precipitate at 0° was probably pure oleic acid, since it melted at 7° C., and must have had an iodine number of about 90 per cent. to make the iodine number of the mixture 109.35.

It is thus plain that the process is not a quantitative separation, but a fractional precipitation, in which the solid acids are precipitated first, then the oleic acid, and lastly the linolic acid.

Though no quantitative separation is effected, the proportion of liquid acids can be calculated from the results. The percentage of liquid acids in the alcoholic filtrate is calculated from the fraction drawn off. This is multiplied by the iodine number of these acids, and deducted from the iodine number of the original fatty acids, and the result represents oleic acid. Dividing this by 0.9 gives the percentage of oleic acid precipitated with the solid acids. Adding this to the liquid acids in the filtrate gives the total liquid acids. Thus, in the case of the last sample, $46.81\% \times 109.35\% = 51.19$. Deducting from 62.57 gives 11.38. Divide by 0.9 = 12.64 oleic acid in the precipitate. Add to 46.81 = 59.45 total liquid acids, the iodine number of which is

$$\frac{62.57}{59.45} = 105.2.$$

The linolic acid may be calculated from the iodine absorption of the liquid acids. 109.35 represents 78.5 oleic acid and 21.5 linolic acid. $21.5 \times 46.81 = 10.06$ linolic acid in original fatty acids.

3. Rose's Process (*Zeit. für Anal. Chemie*, 1886) consists in shaking up an ethereal solution of the fatty acids with litharge. The author substituted petro-

leum spirit for ether, and obtained the following results with the lard fatty acids used in the last experiment :

	Amount per cent.	Iodine No.
Solid acids	44.70	3.02
Liquid acids	55.10	108.66

This method has the disadvantages that it can only be reliable when the fat is quite fresh and great care taken to prevent oxidation. Thus the same sample of lard fatty acids, after standing seven days in a closed jar, gave :

	Iodine No.
Solid acids	10.1
Liquid acids	101.7

The author's general conclusion is that Jean's process, with the addition of taking the iodine absorption of the original acids and of those in solution, will give accurately the percentage of liquid and solid acids in a fat. And although this is too cumbersome for general commercial work, it is as yet the only positive solution of the problem.

C. A. M.

Action of Formalin on Food Stuffs. T. Weigle and S. Merkel. (*Forschungsber.*, etc., 1895, ii., 91; through *Chem. Zeit. Rep.*, 1895, p. 142.)—Milk containing formalin in the proportion of 1 : 5,000 could be preserved at 25° C. for 100 hours; when the proportion was 1 : 10,000 the milk remained good for 50 hours at 25° C. Formaldehyde so changes the albuminoids of milk that they are no longer soluble in a mixture of sulphuric and acetic acids. Moreover, the casein can only be precipitated in thick clots from milk containing formalin, not in the fine flocculent condition characteristic of the casein from normal milk. On this ground alone the addition of formalin to milk may be deemed inadmissible. Formaldehyde renders the albuminoids of milk less digestible, an addition of 1 of formalin to 500 of milk, for example, rendering the casein insoluble in pepsin and hydrochloric acid.

Butters to which formalin has been added increase in acidity very slowly. The saccharification of starch by diastase is favoured by formaldehyde, while alcoholic fermentation is much delayed.

A. G. B.

[Attempts to preserve fish for the market by means of formalin failed in the abstractor's hands on account of the hardening effect of the formaldehyde. This appeared to be due to the coagulation of albuminoids, the samples being thus rendered so hard as to be unsaleable, even by solutions containing 1 part of formalin in 2,000.]

On the Employment of Phenylhydrazine for the Quantitative Estimation of Dextrose, Lævulose, and Saccharose. C. J. Lintner and E. Kröber. (*Zeitschrift für das gesammte Brauwesen*, 1895, No. 19, pp. 153-155.)—The circumstance that glucosazone is almost insoluble in hot water and is thereby distinguishable from the other commonly occurring osazones led the authors to attempt to make use of this property as a means of effecting more reliable quantitative determinations of sugars than is possible by the copper reduction methods or polarization. The process and results may be summarized as follows :

Pure Dextrose.—A solution of dextrose (containing between 0.1 and 0.2 gramme per 20 c.c.) is heated to 100° for one and a half hours with 1 gramme phenylhydrazine and $1\frac{1}{2}$ grammes 50 per cent. acetic acid; and, following the addition of 20 c.c. boiling water, the osazone is collected on a tared filter moistened with hot water, washed with about 60 c.c. boiling water, and dried for three hours at 105–110° C. If the solution is more concentrated the washing of the precipitate is retarded and incomplete; where a larger quantity of wash water is used, a greater proportion of the osazone dissolves—0.1 gramme of dextrose yields 0.1 gramme of osazone; and though this is considerably below the theoretical yield, the results, when the conditions laid down are rigidly adhered to, are remarkably concordant.

Pure Lævulose.—The process is performed in the same manner as for dextrose, but the yield of osazone is somewhat greater (1 : 1.43).

Pure Saccharose.—Previous inversion by dilute HCl and the addition afterwards of sodium acetate is necessary, or the production of osazone is incomplete. The yield is 0.133 gramme of osazone per 0.1 gramme sugar.

Dextrose with Maltose and Dextrin.—Where maltose is present, one and a half hours' heating is requisite to completely form its osazone, and, owing to the difficulty experienced in washing the whole of this out, the dextrose figures are slightly increased (proportion 0.1 : 0.104). In the case of associated dextrin the formation of dextrosazone is retarded, and requires a longer exposure to heat, viz., two hours. The factor is the same as that employed with maltose, but where both are present at the same time the yield is slightly increased (0.1 : 0.106).

The presence of saccharose naturally causes the osazone value for dextrose to come out too high. C. S.

Contribution to the Study of the Ash of Cheese. G. Mariani and E. Tasselli. (*Staz. Sper. Ag. Ital.*, xxviii. 23.)—The authors have estimated the total ash, chlorine, lime, and phosphoric acid in 15 samples of cheese. The amount of salt (calculated from the chlorine) is naturally variable, being dependent on the mode of salting adopted. The proportion of phosphoric acid found was always greater than that necessary to form tribasic calcium phosphate, the proportion varying from 1.07 and 1.08 equivalents P_2O_5 to $1CaO$ in cheese made from sour milk, to 1.56 : 1 in Gorgonzola, 1.67 : 1 in skim-milk cheese, and 1.75 in Edam cheese. The largest quantities of lime and phosphoric acid were found in sheep's-milk cheese and in cheese made from sour milk, whence it follows that acidity does not prevent the precipitation of calcium phosphate with the curds.

The authors attribute the excess of phosphoric acid to the probable presence of acid phosphates. H. D. R.

LITERARY INTELLIGENCE.

A new work, entitled "The Chemistry of Urine; a Practical Guide to the Analytical Examination of Diabetic, Albuminous, and Gouty Urine," by Mr. Alfred H. Allen, is announced by Messrs. J. and A. Churchill. The book occupies about 200 octavo pages, and is illustrated.

THE ANALYST.

AUGUST, 1895.

FEEDING EXPERIMENTS WITH INDIAN PEAS CONTAINING *LATHYRUS SATIVUS*.

By JOHN HUGHES.

(Read at the Meeting, June 5, 1895.)

WITH the assistance of Mr. H. Coleman, of Horton, near Epsom, I have recently been able to carry out, at the farm where I reside, some feeding experiments with Indian peas, as imported, containing 20 per cent. of *Lathyrus sativus* (bitter vetch).

They were purchased of a firm in Mark Lane at 18s. 6d. per quarter, and a mechanical analysis gave the following results:

Indian peas	65.41
<i>Lathyrus sativus</i>	20.63
Foreign seeds	10.81
Mechanical impurities (dirt)	3.15
				<hr/>
				100.00

Separate analyses were afterwards made with the following results:

	Indian peas.	<i>Lathyrus sativus</i> .
Water (lost at 100°C.)	11.66	11.20
Oil	1.87	1.70
Albuminous compounds*	25.06	28.29
Starch, digestible fibre, mucilage, etc.	51.51	49.32
Indigestible fibre	6.97	6.73
Mineral matters†	2.93	2.76
		<hr/>
		100.00
		<hr/>
		100.00

* Containing nitrogen	...	3.96	4.47
† Containing silica	...	0.10	0.10

The peas as delivered, containing the *Lathyrus* and other foreign seeds, were then ground into meal, and the following analysis represents the composition of the mixture employed for the feeding experiments:

Water (lost at 100°C.)	...	12.60
Oil	...	1.60
Albuminous compounds*	...	24.11
Starch, digestible fibre and mucilage	...	48.25
Indigestible fibre	...	7.97
Mineral matters†	...	5.47
		<hr/>
		100.00

* Containing nitrogen	...	3.81
† Containing silica	...	0.10

It will be noticed that the mechanical impurities in the form of dirt have raised the mineral matters to 5.47, while the albuminous compounds only amount to 24.11.

The animals selected consisted of a horse between twelve and fourteen years old, and a three-year-old in-calf heifer.

Commencing on February 27, 1 lb. of the pea-meal mixed with chaffed straw, mangolds and grains, was given every morning to the cow, and 1 lb. mixed with oats and chaff to the horse, for the first fortnight.

No alteration in the general health, or in the character of the dung, was noticeable in either case.

On March 13 the daily allowance of mixed pea-meal was increased to 1½ lb. per day, both for the cow and the horse, and continued till the 27th.

On that date the horse still appeared in his usual health, but during the latter part of the second fortnight there was a slight looseness in the character of the dung, and it was decided to discontinue the pea-meal at the end of the month, and substitute 1 lb. of barley meal, which was done, and by April 3 the dung was again quite normal.

As regards the cow, there was no alteration either in her general appearance or in the dung, and on March 27 the allowance of pea-meal was increased to 2 lb. per day, and maintained regularly till April 10, thus completing a trial of six weeks.

At this date, there being no abnormal appearance in any respect, nor sign of any paralysis, the cow having improved in general health and condition, and as the object of the experiment (namely, to ascertain whether small quantities of *Lathyrus* were or were not poisonous) had been attained, it was decided to terminate the experiment.

There was no reason whatever why the daily allowance of the meal should not have been indefinitely continued, as far as the cow was concerned, and it should be remembered that the 2 lb. of Indian pea meal contained 6½ oz. of *Lathyrus*.

All the recognised authorities state that *Lathyrus* only produces illness after continued or excessive use.

As regards the horse, the 1½ lb. of Indian pea-meal containing 5 oz. of *Lathyrus* given daily for fourteen days, appeared to have caused slight derangement of the digestive organs, the dung being loose and the animal's thirst increased, but there was neither illness nor sign of paralysis; its daily work was performed as usual, and the horse is still in excellent health.

In an alleged case of horse-poisoning at Bristol, it was stated that the horses received 2 lb. per day of Indian peas (roughly crushed) consisting chiefly of *Lathyrus*, and that no signs of illness appeared until after a month had elapsed.

In reporting, therefore, upon any feeding material, we should consider the quantity of the *Lathyrus* present, for it is the quantity that determines the quality. A cake containing 1 or 2 per cent. of sand is not likely to be injurious, but if the sand amounts to 10 or 20 per cent. the cake would very properly be pronounced unfit for feeding purposes.

It is very unfair to condemn a feeding-meal on account of the presence of a few seeds (except in the case of castor).

We should endeavour to make an approximate determination of the quantity of such seed before presuming to condemn the meal as unfit for use.

According to Dr. G. Watts, *Lathyrus sativus*, which originally was indigenous to the North-West Provinces, has spread as a weed seed all over India, being found growing not only with pulse of various kinds, but also with cereals.

Dr. J. Voelcker, in his report on "The Improvement of Indian Agriculture," at page 281, mentions that "*Lathyrus sativus* is one of the impurities found in *clean* wheat as it leaves the threshing-floors of cultivators in the Cawnpore district."

In 1888 I examined a sample of Indian horse gram and detected a foreign seed which I now recognise as *Lathyrus*.

Professor Leather, in his paper, in the *Veterinary Journal* for April, 1885, writes: "So far as we can learn, *Lathyrus* may be used with perfect safety in feeding oxen and sheep, as it does not appear to produce any symptoms in these animals."

PROPORTIONS OF HUSK AND KERNEL.

In order to determine approximately the relation between the husk, the kernel, and the whole seed, ten large and ten small seeds were respectively weighed, coarsely pounded in a mortar, and the husk carefully picked out and weighed.

		Weight of seed.	Weight of husk.	Percentage.
Ten large peas	...	0.895 grammes.	0.131 grammes.	14.63
Ten large peas	...	0.866	0.127	14.66
Ten small peas	...	0.254	0.048	18.89
Ten very small peas	...	0.188	0.033	17.55
Ten large <i>Lathyrus</i>	...	0.838	0.122	14.55
Ten large <i>Lathyrus</i>	...	0.812	0.119	14.65
Ten small <i>Lathyrus</i>	...	0.273	0.058	21.24
Ten very small <i>Lathyrus</i>	...	0.234	0.058	24.78

AVERAGE COMPOSITION.

				Indian pea.		<i>Lathyrus sativus</i> .	
				Large.	Small.	Large.	Small.
Kernel	85.35	81.78	85.40	76.99
Husk...	14.65	18.22	14.60	23.01

The relation of large to small being taken as follows :

				Indian pea.		<i>Lathyrus sativus</i> .	
Large	48.89	64.89	
Small	51.11	35.11	

If, therefore, we assume that 15 parts of husk represent 100 parts of the whole seed, we shall obtain a full approximate estimate of the quantity of *Lathyrus* present in cake or meal.

CHARACTER AND MICROSCOPICAL APPEARANCE OF THE HUSK AND STARCH.

These are shown in the accompanying sheet of photo-micrographs. The testa of both seeds consists of three layers, the two inner ones being so delicate and fragile that they are usually destroyed by the chemicals employed to render the husk transparent.

The external layer is the most important for the purposes of distinction. The most convenient way to prepare it for examination is to boil alternately with acid and soda until cleared, then to thoroughly disintegrate it by grinding between a slide

and cover glass, whereby the fibres which compose it become separated and in a condition to be viewed.

With a one-quarter or one-sixth objective they are seen to be hollow throughout their whole length, and in section hexagonal at one end and stellate at the other; which explains the different appearance presented by the two sides of the husk.

So far the appearance of this outer layer on both the seeds is not very dissimilar. In the middle of the fibres of the *Lathyrus*, however, there is a secondary deposit of spiral tissue like a minute corkscrew, whereas in the fibre of the outer layer of the Indian pea there is no spiral tissue (see Figs. 1 and 2). Consequently a careful examination of this layer is sufficient to distinguish any isolated fragments of the husk that may be found in a feeding-cake or meal.

Of the two inner layers, the middle one is very characteristic in both seeds. In the *Lathyrus* it consists of a thin membrane with a strong resemblance to some patterns of muslin, being composed of numerous circular cells, which average 0.0023 inch in diameter, and which, when once observed, can easily be recognised again (see Figs. 3 and 4).

A surface view of these cells gives a very erroneous impression of their true form, for if viewed edgeways they will be seen to bear a striking resemblance to collar-studs, perforated through their length.

In the Indian pea these cells, from greater compression, lose their circular form and become hexagonal, their size being less—namely, 0.0012 inch diameter (see Figs. 3 and 4). This, together with an absence of the radial dots present in the *Lathyrus*, suffices to distinguish the middle layer in the respective seeds.

The third and innermost layer is exceedingly soft, is destroyed by most clearing agents, and presents no special interest for identification.

STARCH.

It is not easy to differentiate single granules, but when viewed together the points of difference become apparent.

In the *Lathyrus* the granules vary more in size amongst themselves, and are generally rounder than those of the pea, which are more elongated, and frequently show a longitudinal depression well seen with high powers. The relative size of mature grains in the *Lathyrus* is 0.0018 inch diameter, and in the pea 0.0013.

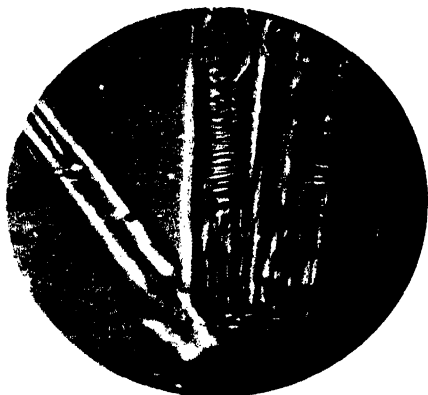
Further, the *Lathyrus* gives a greater play of colour with polarized light than does the Indian pea. When the *Lathyrus* granules are treated with a very weak solution of iodine, the blue coloration is feeble, and produced in a very irregular manner; while in the pea the coloration is much stronger and more uniform, very few granules being left unstained (see Figs. 5 and 6). The iodine solution must, however, be very weak in order to effect this distinction.

In conclusion, I have to acknowledge the valuable assistance of Mr. Albert Ashe in the microscopical work of this paper.

DISCUSSION.

Dr. J. AUGUSTUS VOELCKER said that he had never had any difficulty in distinguishing between a rounded pea like *Pisum arvense* and *Lathyrus sativus*,* which

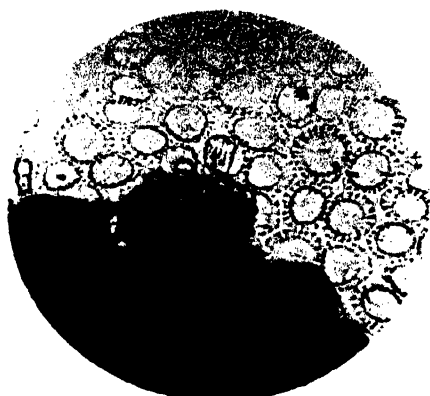
* See ANALYST, xix., 102.



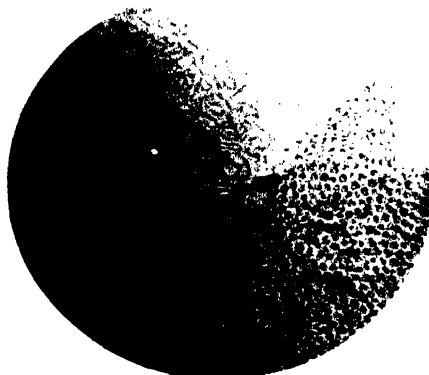
1. X 750.



2. X 600.



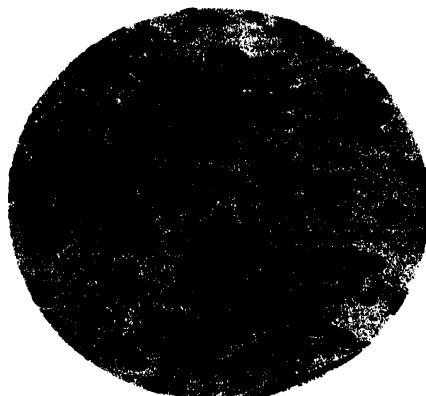
3. X 200.



4. X 200.



5. X 117.



6. X 117.

was totally different in shape and appearance. There was but little difficulty in recognising *Lathyrus sativus*, even when ground up in a cake, as the husk was very characteristic. There was the ovate shape to go by, and also the peculiarly mottled or marbled appearance of the seed, which was completely absent in the common pea.

On the general question, he maintained that the most important point was not the deciding as to whether certain small quantities of *Lathyrus sativus* were in any particular case injurious. In a case where a report had to be made upon a certain small sample, it was quite impossible to give any opinion as to the nature of the whole bulk. It was known to happen frequently that, while one portion of a delivery might contain enough injurious seed to poison a whole herd, another bit from the very same delivery would be quite free from it, and the fact of only a small proportion of injurious matter being found in any particular sample was not evidence that that proportion only was maintained throughout the whole bulk.

He did not contend, and never had contended, that *Lathyrus sativus* was poisonous under *all* circumstances, but that its poisonous properties did actually exist was now recognised. The uncertainty of its action had led him to make inquiries, which had resulted in his being informed by the Director of Botany of Northern India, with whom he had corresponded, of a belief current among the natives that there were *two* kinds of *Lathyrus sativus*, one with and one without poisonous properties, though his informant was unable to say whether this was actually the case or not. The seeds of the two kinds were indistinguishable botanically, though the so-called non-poisonous kind had the seeds somewhat smaller than the other.

Mr. HUGHES, in reply, said he thought it would be very difficult, if not impossible, to recognise *Lathyrus sativus* without the aid of the microscope when incorporated in a feeding meal or cake, as it would in all probability be ground very finely.

He was able to confirm what Dr. Voelcker had said as to the existence of apparently two kinds of *Lathyrus sativus*, from information which he had received from Dr. King, of the Botanical Gardens, Calcutta. The fact of the seeds being indistinguishable botanically was not surprising, as a botanist really required the whole plant for purposes of identification, the seeds alone being of but little use. It was well known that in India the crop was grown by the natives as food for themselves and their animals, in spite of the fact that they were aware of its occasionally injurious properties, which, however, they endeavoured to counteract by cooking the peas, and by using them in moderation. It is only when compelled by famine to use the seeds of *Lathyrus sativus* as the exclusive daily food that the natives experience disastrous results. Mr. Arthur Church, of the Botanical Gardens, Oxford, believes that much confusion at present exists in regard to the different species of *Lathyrus*, and that a more perfect identification would determine which species were dangerous and which were harmless.

PSEUDO-WOOLLEN FABRICS.

By E. G. CLAYTON.

(Read at the Meeting, June 5, 1895.)

A CONSIDERABLE amount of attention was directed a few months since to some published statements referring to the material known as "flannelette." In common, no doubt, with many other persons, the writer was previously under the impression that flannelette consisted of wool and cotton in about equal proportions. To a casual observer, much of the flannelette sold would seem to be softer and warmer than many flannels.

It is likely, therefore, that for some years past flannelette has been purchased frequently for the clothes of delicate persons and children, under a mistaken notion that it was the most suitable material for that purpose; instances of this are known to the writer.

The published statements alluded to above pointed to the probability that an investigation of samples of flannelette of different values, and procured at various times and places, would be of some interest.

The results of a microscopical examination of ten samples of flannelette, obtained in various neighbourhoods, and supplied at different prices, showed that six of them were all cotton, and that four of them did not contain more than 5 per cent. of wool.

Of nineteen samples of material sold as "sanitary flannel" similarly examined, sixteen were found to be all wool, one nearly all wool, one chiefly cotton, and one all cotton.

Eighteen samples of "flannel" were found to consist entirely of wool.

NOTE ON ADULTERATED SANDAL-WOOD-OIL.

By T. H. PEARMAIN AND C. G. MOOR, M.A.

(Read at the Meeting, June 5th, 1895.)

WE have recently met with some gelatine capsules labelled and sold as "pure sandal-wood-oil capsules," the contents of which, on examination, proved to be castor-oil mixed with a small quantity of an unsaponifiable oil which we have not been able to identify. The oil had the characteristic taste and smell of castor-oil. It gave the following figures on analysis:

Specific gravity at 15.5° = .9633.

Iodine absorption = 88.0 per cent.

Unsaponifiable matter = 5.0 per cent.

Valenta test (using 84 per cent. acid) turbid at 100° C.

Miscible with glacial acetic acid.

Miscible with 3 volumes of rectified spirit.

Saponification value = 17.0 per cent. KHO

(Corrected for unsaponifiable matter = 17.9 per cent. KHO).

Rotation for 100 mm. at $15.5^{\circ} = +8.0^{\circ}$.

Bromide of tin test = bright green.

Fatty acids:—

Solidifying-point, 0° C.

Melting-point, 13° C.

Iodine absorption, 90.1 per cent.

The somewhat high figures obtained in the case of the iodine absorption and rotation we attribute to the presence of the unsaponifiable oil. The highest dextro-rotatory castor-oil we have examined had an activity of 4.7° .

The unsaponifiable matter gave the green colour with the stannic bromide test to a much more marked extent than the original oil, thus proving this body to be the cause of the green coloration.

The stannic bromide test is performed as follows: To three drops of the oil under examination are added three or four drops of freshly-made stannic bromide containing a little free bromine,* the mixture well stirred, and the colour produced after two or three minutes noted. Genuine sandal-wood-oil gives a blood-red colour, the mixture becoming almost dry and solid after one hour. Cedar-oil, the most common adulterant of sandal-wood-oil, gives a purplish colour. A mixture of the two does not solidify on standing.

On account of the difficulty of obtaining a sufficient quantity of material, we have been obliged to omit many estimations, the results of which would have been of interest.

We found it necessary to dilute the acetic acid before applying the Valenta test, sandal-wood-oil being miscible with glacial acetic acid. 84 per cent. acid was found to be the most convenient strength.

The following are some results obtained on some genuine sandal-wood and cedar oils:

Sampl	Oil.	Source.	Sp. Gr. at 15.5° C.	Iodine Absorption.	Rotation 100 mm. at 15.5° .	Valenta Test (84% Acid).	Bromide of Tin Test.
A	Sandal-wood	Indian	0.9815	226.0	L 13.84°	60°	Blood-red
B	"	"	0.9792	225.0	L 15.21°	50°	"
C	"	"	0.9798	246.5	L 15.55°	52°	"
D	"	"	0.9792	244.1	L 15.70°	47°	"
E	"	"	0.9840	215.3	L 19.53°	—	"
F	"	Australian	0.9496	239.5	R 6.19°	50°	"
G	"	—	0.9495	228.6	R 4.82°	52°	"
H	Cedar	—	0.9819	200.4	L 39.0°	above 100°	Purple
I	"	—	0.9840	241.5	L 26.0°	"	"
J	"	—	—	—	L 30.0°	"	"

* Made by allowing dry bromine to fall drop by drop on granulated tin contained in a flask kept cool by immersion in cold water, until the product has a red colour indicating excess of bromine.

NOTE ON A SAMPLE OF ADULTERATED COFFEE.

BY T. H. PEARMAIN AND C. G. MOOR, M.A.

(Read at the Meeting, June 5, 1895.)

WE have recently obtained a small sample of adulterated coffee, which we are informed is about to be placed on sale in this country. As the results obtained on examining the sample are somewhat interesting, we thought this note would be worth communicating to the society. The sample gave on analysis the following figures :

						Per cent.
Moisture	3.2
Fat	12.3
Total ash						3.4
Insoluble ash						0.7
Soluble ash						2.7
Nitrogen		2.7

A filtered 10 per cent. decoction had the same specific gravity as genuine coffee, namely, 1009.5. After some little trouble we found the sample to consist of exhausted coffee, together with ground acorns.

We were unable to isolate any appreciable quantity of caffeine from the sample. The microscopical appearance of the sample is remarkable, and would render its detection easy even if mixed in small proportion with genuine coffee. In addition to the well-known coffee structures are a number of starch-containing cells, arranged not unlike the heads of Indian corn, together with cellular membrane-containing spiral vessels, which we found to be characteristic of ground acorns.

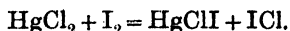
On the Hübl Method of Iodine Absorption. Julius Ephraim. (*Zeit. für angewand. Chemie*, Heft ix., 1895, pp. 254-259.)—Since the Hübl solution, when titrated with thiosulphate, requires a much larger quantity of the latter after the addition of KI, the conclusion is arrived at that a substance capable of liberating I from KI must be present. This substance is probably a compound of iodine and chlorine formed by the action of the iodine on the HgCl_2 . On mixing the two portions of the Hübl solution it appears to be formed at once. Thus the author obtained the following results :

25 c.c. Hübl Solution required.					$\text{Na}_2\text{S}_2\text{O}_8$
I. Immediately after mixing :					c.c.
(a) Without KI	14.30
(b) With KI	51.10
II. After twenty minutes :					
(a) Without KI	14.45
(b) With KI	50.25
III. After four hours fifty minutes :					
(a) Without KI	13.25
(b) With KI	48.95

(1 c.c. $\text{Na}_2\text{S}_2\text{O}_8 = 0.01223$ gramme I.)

He next shows that both iodine monochloride and iodine trichloride can be titrated directly with $\text{Na}_2\text{S}_2\text{O}_3$, and proves that the latter cannot be the compound present in the Hübl solution, since results altogether discordant are obtained when it is tried with commercial oleic acid.

Hübl's requirement, that for two atoms of iodine there must be at least one molecule of HgCl_2 , may be explained by the following equation, on the assumption that iodine monochloride is the compound formed :



To make a solution corresponding to the strength of the Hübl solution this would give 16.25 grammes of ICl per litre, and this proportion is used by the author for his solution.

Since iodine monochloride as met with in commerce is seldom quite pure, it is not sufficient merely to ascertain the amount of real ICl which it contains by titration with the addition of KI , but also without, so that the amount of I which the impurities in the ICl are capable of liberating in the substance itself may be ascertained.

The method, which is carried out in the same way as the Hübl process, gave the following results :

Oil.		Iodine Number with Iodine Monochloride.		Hübl Iodine Number.
Oleic acid, I.	82.7	...	82.84
„ II.	82.77	...	
„ III.	82.5	...	
„ IV.	82.74	...	
Linseed oil, I.	179.7	...	179.9
„ II.	177.7	...	
Olive oil, I.	83.9	...	84.3
„ II.	84.00	...	
Poppy oil	81.13	...	81.9
Sesame oil, I.	137.1	...	137.3
„ II.	137.1	...	
Earthnut oil, I.	110.6	...	111.1
„ II.	109.7	...	
Castor oil, I.	83.33	...	
„ II.	83.09	...	
Almond oil, I.	96.00	...	
„ II.	96.29	...	

From these figures the author concludes that a solution of iodine monochloride in alcohol may be substituted for the Hübl solution. The advantages are that it is more readily prepared, being easily soluble in alcohol, and when made is ready for immediate use. Moreover, it can be made in much more concentrated solution, and, lastly, is cheaper. Its chief disadvantage is that it is not yet made in a state of sufficient purity, and, therefore, each fresh quantity obtained must be titrated, as mentioned above.

If the ICl solution, after acting on the oil, be titrated without previous addition of KI , a new value is obtained, which the author calls the "chlor-iodine number." In titrating, the thiosulphate is added until the liquid, which had become brown with separated iodine, again becomes yellow. The solution is then diluted, starch added, and the titration completed.

The reaction taking place is :



in which 161.5 parts of $\text{ICl} = 316$ of $\text{Na}_2\text{S}_2\text{O}_3$. The following chlor-iodine numbers were obtained :

			I.		II.
Earthnut oil	56.6	...	55.91
Linseed oil	77.03	...	76.7
Sesame oil	...	*...	65.3	...	65.0
Rape-seed oil	50.4	...	50.06
Poppy oil...	43.4	...	43.4
Castor oil...	44.24	...	43.88

A few experiments tried with iodine bromide also gave concordant results :

			I.		II.	III.
Almond oil	58.5	...	58.6	
Olive oil	52.6	...	52.81	52.24
Earthnut oil	62.9	...	63.29	

The solution of IBr contained 20.7 grammes per litre.

C. A. M.

The Influence of Temperature on the Elaidin Reaction. A. P. Lidow. (*Pharm. Zeit. f. Russland*, xxxiv., 105-106, through *Chem. Centralblatt*).—By the action of nitrous oxides on oleic acid at 0°C . a substance was produced which melted at 42°C ., while there was an increase in weight of 1 per cent. At $80-85^\circ$ there was an increase of weight of 16.6 per cent., and the iodine number of the product was only 9 per cent., whereas that of the oleic acid used was 88.6 per cent. It thus appears that at this temperature the free affinities of the unsaturated acids are satisfied. By long-continued action of the nitrous oxides the elaidic acid formed becomes fluid.

C. A. M.

Action of Alcoholic Soda Solution on Albumin and on Glue-yielding Substances. W. Fahrion. (*Chem. Zeit.*, 1895, xix., 1000-1002).—The author has already shown (*Chem. Zeit.*, 1893, xvii., 434) that unsaturated fatty acids easily undergo polymerization, and that the polymerides are equally easily broken up by alcoholic soda solution; since albuminoids and gelatinoids have been regarded as polymerization products, it appeared to be of interest to study their behaviour with the same reagent.

Ten grammes of pure hide-powder were digested on the water-bath with 50 c.c. of alcoholic soda solution (8 per cent.), whereupon nearly all of it dissolved, a little ammonia being at the same time evolved. The alcohol was evaporated, the gelatinous residue dissolved in hot water, and the solution cooled and filtered. The filtrate was heated, acidified with hydrochloric acid—which expelled some carbonic acid and a little sulphuretted hydrogen—cooled, and filtered. The yellow filtrate was evaporated to dryness, the residue was dried at 110 to 120° until constant in weight, and extracted with hot absolute alcohol; the alcoholic solution left a sticky red-brown syrup when evaporated.

This product is nearly ash-free and quite sulphur-free; it cannot be crystallized. It has a sour odour and a disagreeable, bitter taste. It is insoluble in ether and in

light petroleum, but dissolves easily in water and in alcohol. When heated for some time it swells up, but does not apparently decompose; when cooled again it becomes brittle and easily powdered, but the powder is extremely hygroscopic. The substance is undoubtedly an acid, for it liberates carbonic acid from sodium carbonate in the cold, but its salts appear to be soluble, for its aqueous solution yields no precipitate.

An analysis of the acid, dried at 110° , indicated the formula $C_8H_{16}N_2O_6$ for the compound, but the author is disposed to regard it as still containing a molecule of water, and as being in reality identical with the acid obtained by Schutzenberger by heating albumin with baryta under pressure, and called by him proteic acid, $C_8H_{14}N_2O_5$. Towards the carbonates of the alkaline earths the acid behaves as though it were monobasic, but when it is heated with the alkaline earths themselves it behaves as a dibasic acid; this observation leads the author to conclude that proteic acid is a lactonic acid, which yields its appropriate salts when it reacts with carbonated alkalis, but salts of a dibasic acid containing one more molecule of H_2O when heated with caustic alkalis.

The same acid was obtained by the similar treatment of glue, degreased ox-flesh, casein, human hair, horn, wool, and silk, indicating that the proteids in general are not aldehydic in constitution, as supposed by Loew, but lactonic.

More important from the point of view of this journal are the possible applications of this reaction of proteids in analysis.

To distinguish wool and silk from cotton, a sample of the fabric is warmed with 8 per cent. alcoholic soda solution; wool and silk dissolve with the exception of a little dark flocculent matter, but cotton is not attacked. Starch yields a black substance when treated with the alcoholic soda solution, but this is insoluble in alcohol, so that a quantitative estimation of albuminoids in presence of starch will probably be possible by this method.

Most oils and fats contain a small percentage of albuminous substance, and when they are saponified with alcoholic soda, this albumin is converted into proteic acid, which remains dissolved. When an excess of acid is added, and the liquid is shaken with petroleum ether for the purpose of separating the hydroxy-fatty acids (*loc. cit.*), which are insoluble in this solvent, the proteic acid also remains undissolved, and can be detected in the hydroxy-acids by means of its nitrogen. It is the presence of this proteic acid which has misled Jahoda and Simand in their investigation of dégras; these chemists have maintained that a peculiar nitrogenous constituent—the “dégras-former”—is essential to genuine dégras, and can be recognised by its insolubility in petroleum ether. The author has shown, however, that by shaking the dégras with ether and water before saponification, the portion of the fatty acids which is insoluble in petroleum ether is free from nitrogen, consisting, in fact, of pure hydroxy-fatty acids. The method by which dégras is obtained necessitates the presence of a small quantity of hide-fibre, which will, of course, yield proteic acid when the dégras is saponified; thus the presence of nitrogen in that portion of the fatty acids of dégras which is insoluble in petroleum ether is not *essential* to genuine dégras, although it is indicative that the material has been obtained from chamois leather.

The author submits the following scheme for the analysis of chamois leather and of glacé kid:

Two portions of 5 grammes of the finely-divided sample are weighed; the one is dried at 110° to 120° for the estimation of water, and subsequently ashed for the determination of mineral matter; the other is saponified in a porcelain dish with 8 per cent. alcoholic soda solution; the alkaline solution is washed into a separating funnel, acidified with hydrochloric acid, and when cold shaken with ether. The solid hydroxy-acids—the higher oxidation products of the jecoric acid which is characteristic of the fish oils—are thus left undissolved; they are dissolved in warm alcohol, and, after this has been evaporated, weighed. The ethereal solution contains the unsaponifiable matter, the fatty acids, and the liquid hydroxy-fatty acids. It is evaporated, and the weighed residue is treated with petroleum ether, which leaves the hydroxy-acids undissolved. The petroleum ether solution is shaken in a separating-funnel with aqueous alcoholic soda solution; the fatty acids are thus extracted, the unsaponifiable matter (cholesterin) remaining dissolved in the petroleum ether. The alkaline solution of the fatty acids is evaporated to dryness, the residue dissolved in hot water, the solution decomposed with hydrochloric acid, and the fatty acids shaken out with petroleum ether (distilling completely at 75°).

The following table gives some analyses made according to this scheme. The small quantity of substance insoluble in the original alcoholic soda solution has been neglected, and the hide substance has been calculated by difference:

	Water, Per Cent.	Ash, Per Cent.	Unsaponifi- able matter, Per Cent.	Fatty Acids, Per Cent.	Liquid Hy- droxy-acids, Per Cent.	Solid Hy- droxy-acids, Per Cent.	Hide Substance, Per Cent.
Chamois leather (Sheep), I. ...	18.66	8.28	0.49	4.15	0.37	0.53	67.52
" " " II. ...	17.95	1.88	0.10	1.17	Trace	0.69	68.71
" " " III. ...	17.60	4.38	0.30	3.10	0.28	1.00	73.84
" " (Doe) ...	15.15	6.03	0.49	4.16	0.45	1.37	72.35
" " (Goat) ...	15.13	3.83	3.03	0.28	0.61	0.56	76.51
" " (Buffalo) ...	20.54	3.21	0.10	0.46	0.11	0.56	75.02
Glacé kid ...	11.69	7.12	3.66	6.58	0.66	0.75	69.54

It is particularly noticeable from these figures how very little fatty matter serves to convert skins into chamois leather. The author is disappointed to find that the proportion of fatty acids present is, as a rule, much greater than that of the hydroxy-acids, since, according to his theory, the curing of the hide-fibre depends on its combination with hydroxy-fatty acids; he has not yet, however, satisfied himself that the fatty acids are not merely derived from the natural fat of the skin.

A. G. B.

The Examination of Pepper. W. Busse. (*Mitth. Gesundheitsamt.*, 505, through *Zeit. angewand. Chem.*)—In the author's opinion most of the analytical methods for the valuation of pepper, such as the determination of the moisture, ethereal oil, mineral matter, alkalis, and phosphoric acid, are inconclusive, since these constituents are common to the seed and the husk. The value of the cellulose and dextrose estimations must also be called in question. On the other hand, the brown colouring substances are only found in the husk, and their quantitative estimation

will show the value of the pepper. To isolate them the following method is proposed. Five grammes of the sifted and dried pepper are treated with boiling absolute alcohol. The residue, after being freed from alcohol in the drying-oven, is ground up with a little water in a basin, and then washed into a flask with 50 to 60 c.c. of boiling water. Twenty-five c.c. of a 10 per cent. solution of soda are added, and the flask is warmed on the water-bath for five hours with constant shaking. Concentrated acetic acid is next added, until the liquid is only feebly alkaline, then 250 c.c. of water, and the flask is well shaken. After twelve hours the liquid is filtered with the aid of a suction-pump.

To 50 c.c. of the filtrate concentrated acetic acid is added to acid reaction, followed by 20 c.c. of a 10 per cent. solution of lead acetate in dilute acetic acid. After mixing, the liquid is diluted to a 100 c.c. with water, well shaken and filtered. Ten c.c. of the filtrate are decomposed with 5 c.c. of H_2SO_4 (1 : 3) and 30 c.c. of alcohol, the precipitate filtered after some time, washed with alcohol, and the lead sulphate ignited and weighed in the usual manner, and the amount of lead calculated.

The amount of lead (in grammes) which has been obtained by the process from 1 gramme of dried pepper, may be described as the "lead number." The figures given by the different kinds of pepper are :

				Lead Number.	
White pepper	0.006	to 0.027
Black pepper	0.054	„ 0.075
Husks	0.129	„ 0.157
Pepper dust	0.109	„ 0.122

The author concludes that this method, taken in conjunction with the determination of the ash and sand and the microscopical examination, should be of great assistance in estimating the value of pepper. C. A. M.

Simple Method of Detecting "Poivrete" in Ground Pepper. D. Martelli. (*Staz. Sper. Ag. Ital.*, xxviii., 53.)—Several methods other than microscopical ones have been proposed for the detection of "poivrete" (ground olive-stones). Thus, Gillet (*Bull. Soc. Chim.*, 50, 178) used tincture of iodine (6.5 gr. I in 120 c.c. 90 per cent. alcohol), which coloured pepper brown or dark chestnut, while olive-stones became bright yellow. Chevreau (*Rep. Pharm.*, 1889, p. 203) does not consider this reliable, and employs a mixture of 1 part aniline with 3 parts acetic acid, which colours "poivrete" a light yellow. Juneau (*Journ. Pharm. Chim.*, 1889, p. 442) used a mixture of 5 grammes iodine in 100 c.c. of a mixture of alcohol and ether, the colours produced being the same as with Gillet's test. Pabst (*La Pratique des Essais Commerciaux—Matières Organique*, Halphen, p. 21) tests by adding a little of a solution of dimethyl-para-phenylene-diamine; after a short time the ligneous particles foreign to pepper become carmine-red, while pepper at most is superficially tinged pink.

The author proposes the following test: Digest for two or three days 1 gramme of phloroglucol in 50 or 60 c.c. of hydrochloric acid sp. gr. 1.1, and decant the clear solution. To about $\frac{1}{4}$ gramme of the sample of pepper add enough of the reagent

to cover it, and heat cautiously till fumes of hydrochloric acid begin to come off. "Poivrette" and like substances (*e.g.*, ground-up shells of almonds, walnuts, nuts, etc.) give a very intense cherry-red colour, which is sharply distinguished by the naked eye from the yellow or faintly red-brown colour of the pepper. On adding to the mass a little water, and decanting the liquid, a violet-red powder is left, which consists almost entirely of "poivrette," etc., stained by the reagent.

H. D. R.

A Comparative Examination of the Constitution of Different Meat Extracts.

A. Stutzer (*Zeit. angew. Chem.*, 1895, p. 157).—The meat extracts most frequently met with are those of the Liebig and Kemmerich Companies and the various Bovril preparations. According to the author's analysis, these contain:

	Liebig Extract.	Kemmerich Extract.	Bovril Fluid Beef.	Bovril Fluid Beef seasoned.	Bovril for Invalids.	Bovril Beef Jelly.	Bovril Lozenges.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Water	17.72	16.54	29.14	44.42	28.13	89.15	9.47
Sodium Chloride ...	3.11	4.15	14.12	10.72	4.57	0.26	1.63
Other Salts	19.63	17.96	3.38	7.60	11.50	1.04	5.71
Organic Matter ...	59.54	61.35	53.36	37.26	55.80	9.55	83.19
The most important constituents of the organic matter are those containing nitrogen. Nitrogen was present in the form of—							
(a) Albumose Peptone	0.56	1.24	1.23	0.34	1.26	0.16	2.06
(b) Pancreas Peptone...	2.72	2.38	3.36	1.39	3.36	0.48	6.06
	3.28	3.62	4.59	1.73	4.62	0.64	8.12
(c) Flesh bases and de- composition products soluble in alcohol ...	4.05	3.69	1.06	1.16	1.78	0.21	0.55
(d) Ditto, insoluble in alcohol	1.34	1.25	1.16	0.89	0.82	0.20	1.16
	5.39	4.94	2.22	2.05	2.60	0.41	1.71
(e) Albumin	0.12	0.09	0.31	0.08	0.24	—	0.42
(f) Muscular Fibre ...	—	—	0.73	0.90	0.70	—	0.57
	0.12	0.09	1.04	0.98	0.94	—	0.99
(g) Gelatin	0.04	0.05	0.09	0.09	0.15	0.29	0.70
(h) Ammonium Salts ...	0.48	0.46	0.31	0.27	0.38	0.12	0.42
	0.52	0.51	0.40	0.36	0.53	0.41	1.12
Total Nitrogen...	9.31	9.16	8.25	5.12	8.69	1.46	11.94

With regard to this table, the author remarks:

I. The value of a meat extract as a food material depends on the amount of peptone present; and of the two kinds referred to under (a) and (b), the albumose

peptone probably possesses greater nourishing power than the other. Calculating for 1 part of nitrogen $6\frac{1}{4}$ parts of peptone, the amount of the latter present in the various preparations is as follows :

	Per cent.
Liebig's Meat Extract	20.50
Kemmerich's Meat Extract	22.62
Bovril Fluid Beef	28.68
" " (seasoned)	10.81
" for Invalids	28.87
" Beef Jelly	4.00
" Lozenges	50.75

Thus the statement that 1 part of bovril fluid beef seasoned contains a greater amount of nourishing material than 50 parts of any other meat extract is quite inadmissible.

II. The value of a meat extract as a stimulant (apart from the action of the salt) depends especially on the quantity of the substances classed under (c) and (d). In this respect the Liebig and Kemmerich preparations take the first place.

III. In addition to albumin, the bovril preparations contain considerable quantities of muscular fibre (up to 6 per cent.).

IV. Gelatin should be regarded as a worthless constituent, and should be removed from meat extracts as completely as possible.

C. A. M.

Determination of Glycerol in Wines, and Indirect Estimation of Mannitol.
G. Mancuso-Lima and G. Sgarlata. (*Staz. Sper. Ag. Ital.*, xxviii., 236.)—The authors find that the process of Oliveri and Spica (*Gazzetta*, xx., 734), who distil the glycerol under reduced pressure at 200°, and titrate with $\frac{N}{10}$ permanganate, too long and tedious when many samples are to be examined.

They base their process on the fact that ammoniacal lead hydroxide, recently prepared, completely precipitates both glucose and mannitol, and that the glycerol can be titrated by permanganate in the filtrate after removing the lead and adding sulphuric acid.

1 c.c. of normal permanganate is equal to .01 gramme glycerol, .0081 gramme glucose, and .0089 gramme mannitol.

The direct estimation of glycerol is performed by concentrating 25 c.c. of the wine to a syrup on the water-bath, and precipitating in the cold by ammoniacal hydroxide of lead (obtained by adding a slight excess of basic acetate of lead, and then an excess of ammonia); the liquid is filtered rapidly through a filter, contained in a funnel, over the top of which is placed another funnel connected with a tube containing caustic potash, and the filtrate and washings collected in a beaker, acidified with concentrated sulphuric acid, and the sulphate of lead separated by filtration; the solution is brought to the boil, and titrated hot with normal permanganate.

By precipitating with basic acetate of lead, and proceeding as before, the glucose and mannitol (if present) are left in solution, and are titrated with the glycerol with permanganate; by estimating the glucose by Fehling's solution, and subtracting the amount of permanganate equal to this and to the glycerol, from the total used, the remainder is equivalent to the mannitol.

H. D. R.

The Detection of Salicylic Acid in Wine. M. Spica. (*Staz. Sper. Ag. Ital.*, xxviii., 246.)—The reaction between ferric chloride and salicylic acid, after shaking out with ether, will not detect less than .003 gramme per litre; ether, chloroform, and carbon bisulphide all extract tartaric acid, tannin, and acetic acid from the wine, which interfere with the salicylic-reaction, and render the test less delicate. By evaporation of the ether-extract, and extraction of the residue with petroleum ether, the test is more delicate, .002 gramme being detected.

The author heats the ether extract with nitric acid, by which means the salicylic acid is transformed into trinitrophenol (picric acid). He proves that genuine wines give no extract to ether from which picric acid is formed, and that saccharine (Fahlberg) does not yield picric acid. The delicacy of the reaction is such that .0002 gramme salicylic acid per litre can be detected.

The test is performed thus: 10 c.c. of wine are acidified with a few drops of hydrochloric acid, and shaken with an equal volume of ether; half the ether is removed and filtered (this is not absolutely necessary); the ether is evaporated, and one drop of nitric acid is added, and the mixture cautiously heated over a flame; a few drops of ammonia are added, and then 1 c.c. of water and a piece of wool, previously treated with potash solution, and then with ether to remove fat; the solution is warmed, and the wool, after washing and drying between blotting-paper, is examined; it is dyed yellow, if salicylic acid is present. By comparison with a set of standard wools, prepared from wines to which known quantities of salicylic acid have been added, an estimation may be made.

H. D. R.

Estimation of Potassium Sulphate in Wine. L. Hugouneng. (*Journ. Pharm. Chim.*, 1895 [6], i., 349; through *Chem. Zeit.*)—The author notes that the percentage of sulphuric acid (weighed as barium sulphate) found in a wine depends on whether the estimation has been made in the wine itself or in the aqueous extract of the ignited total solids. The ash yields a higher percentage than does the wine itself. This discrepancy is probably due to the existence of sulphur compounds other than potassium sulphate in the wine, which become potassium sulphate when the residue is ignited. It will be found that this difference between the sulphuric acid in the wine and in the ash of the wine is characteristically high (0.2 gramme per litre) in wine from dried fruits, a fact which proves useful in the diagnosis of a particular sample.

A. G. B.

The Determination of Tannin in Wine. A. Vigna. (*Staz. Sper. Ag. Ital.*, xxviii., 19.)—Two modifications of the Carpené-Lowenthal method (precipitation with ammoniacal zinc acetate, heating to drive off excess of ammonia, and titration of the precipitate after dissolving in sulphuric acid by permanganate) are proposed. In the first, 25 c.c. of an 8 per cent. solution of zinc acetate are added to 50 c.c. of wine, and neutralized exactly with dilute ammonia, and the precipitate collected on a plain filter and washed four times with cold water. The filter is then placed on a glass plate, and the precipitate washed off by a stream of water from a wash-bottle; 20 c.c. dilute sulphuric acid (1 part acid to 4 parts water) and 20 c.c. of indigo

solution are added, and the titration is performed in the usual manner with permanganate. In the second modification, 40 c.c. of an 8 per cent. solution of potash alum are used in place of the zinc acetate, the remainder of the process being the same as before.

The author shows that both modifications give the same results as the Carpené-Loewenthal method with wines, and theoretical results with pure tannins. He prefers the alum method, as being the more expeditious.

H. D. R.

Detection of Fluorine in Wine. G. Nivière and A. Hubert. (*Monit. Scient.*, 1895 [4], ix., 324; through *Chem. Zeit.*)—100 c.c. of the wine are made feebly alkaline with ammonium carbonate, the solution is boiled, and 2 to 3 c.c. of a 10 per cent. solution of calcium chloride are added. After boiling for a few minutes, the liquid is filtered, the filter ashed, the ashes mixed with a third of their weight of precipitated silica, and the mixture transferred to a test-tube, together with a mixture of equal parts of fuming sulphuric acid and concentrated sulphuric acid (66° Bè). The test-tube is fitted with a cork, which carries a U-tube 4 millimetres in diameter, and having three bulbs (7 to 10 millimetres diameter) blown in it. In the centre bulb one drop of water is placed. The test-tube is now heated, whereupon any fluorine will be evolved as silicon tetrafluoride, which will be decomposed by the drop of water, yielding the usual gelatinous silica and solution of hydrofluosilicic acid. In the event of mere traces of fluorine being present, no visible separation of silica in the water-drop will be noticed; in this case the U-tube must be washed out with alcohol and the washings evaporated to dryness, when the silica will be detected. A blank test must be made with the filter-paper, and great stress must be laid upon the fine state of division of the silica and upon the prescribed strength of the sulphuric acid.

For quantitative estimations the U-tube is washed with water, the washings are filtered, and to the filtrate is added a solution of potassium acetate in dilute alcohol; the precipitated potassium silicofluoride is filtered, washed with dilute alcohol (1 : 1), dried, and weighed. The factor 0.5177 will convert the weight into the corresponding weight of fluorine. One litre of wine will be necessary for a quantitative determination. The author has detected the fluorine corresponding with 1 gramme of ammonium fluoride in one hectolitre of wine with rapidity and certainty by this method.

A. G. B.

Detection of Fluorine in Beer. R. Hefelmann and P. Mann. (*Pharm. Centr.-H.*, 1895, xvi., 249; through *Chem. Zeit.*)—A fluoride which has been added to finished beer as a preservative may be detected as follows: To 500 c.c. of the beer, freed from carbonic acid—either by exposure in a thin layer, or by heating at 40°—1 c.c. of a mixture of equal volumes of calcium chloride solution (10 per cent.) and barium chloride solution (10 per cent.) is added; this is followed by 0.5 c.c. of acetic acid (20 per cent.) and 50 c.c. of alcohol (90 per cent.). The liquid is allowed to remain for twenty-four hours in the cold, in order that the precipitated calcium fluoride and barium silico-fluoride may settle; it is then filtered through a small filter, the last traces of the precipitate being either washed out of the beaker by

means of the filtrate, or wiped out by means of filter-paper. The precipitate and filter are dried without being washed, and transferred to a platinum crucible (20 c.c.); 1 c.c. of strong sulphuric acid is added, the crucible is covered with a clock-glass (which has been waxed and then marked with a stylé), this is filled with water, and the crucible is heated at 100° for two hours. By this method so little as 0.7 milligrammes of fluorine in 100 c.c. of beer can be detected with certainty.

A. G. B.

The Detection of Bloodstains in Forensic Cases. F. Gantter. (*Zeit. für anal. Chem.*, 1895, *Zweites Heft*, pp. 159-160.)—The detection of bloodstains on rusty iron is a matter of extreme difficulty, owing to the fact that in most cases it is impossible to obtain hæmine crystals from the blood which has been made insoluble by the iron oxide. Very frequently, too, it is of the greatest importance to prove the complete absence of blood.

The behaviour of the blood towards hydrogen peroxide is a valuable means for the latter purpose. The slightest trace of blood-substance brought into contact with a drop of this reagent causes an immediate evolution of oxygen, so that the drop in a very short time becomes changed into a white froth.

The best method of observing the reaction is as follows: A drop of the solution of the suspected blood-material, or where a solution is not possible (as in the case of spots on rusty iron), a small piece of the rust scraped off, is placed on an object-glass with a piece of black paper underneath. After moistening with a drop of water made very feebly alkaline a drop of hydrogen peroxide is added, and when the slightest trace of blood-substance is present numerous comparatively large bubbles of gas are developed, which after a short time unite, forming a fine snow-white scum, which remains for some hours. It is characteristic that the scum forms from the outside of the drop inwards, so that it is surrounded by a ring of clear fluid. In the case of rust containing blood, the development of gas does not proceed from all the particles of rust but only from those to which blood is attached.

It is impossible to mistake this appearance for that formed by air-bubbles. On moistening the rust with the alkaline water numerous air-bubbles are frequently formed, but these are quickly dissipated by touching them with a thin glass rod before adding the hydrogen peroxide.

If the reaction does not occur, it is certain that the rust contains no blood; but, unfortunately, its occurrence cannot be regarded as proof positive of the presence of blood, since other animal fluids (*e.g.*, pus) behave in a similar way towards hydrogen peroxide.

Apart from its value as a negative proof, the reaction is also of use in testing whether crystals supposed to be hæmine crystals really are so. The age of the spots on the rust appears to make no difference. Stains six months old responded to the test as sharply as when fresh.

C. A. M.

Examination of Medicaments containing Iodine. G. Deniges. (*Journ. Pharm. Chim.*, 1895 [6], i., 354; through *Chem. Zeit.*)—Since a large number of organic iodine preparations easily yield their iodine as alkali-metal iodide, it is possible

to assay them by the usual methods for titrating iodine. Iodoform is decomposed in a test-tube by heating it for a short time with 1 c.c. of nitric acid and 2 c.c. of hydrochloric acid; 10 c.c. of water are added and 2.5 c.c. of sodium bisulphite solution (36.4° B). The iodine having been thus completely converted into hydriodic acid, 10 c.c. of ammonia are added, and the whole is made up to 100 c.c. Fifty c.c. are then mixed with 15 c.c. of ammonia and 25 c.c. of $\frac{N}{10}$ silver nitrate solution, made up to 100 c.c. and filtered. Fifty c.c. of the filtrate are mixed with 50 c.c. of water and 10 c.c. of $\frac{N}{10}$ potassium cyanide solution; a few drops of potassium iodide solution (20 per cent.) are next added, and the liquid titrated with $\frac{N}{10}$ silver nitrate solution until a permanent turbidity is produced.

Di-iodoform is decomposed by heating it in a long test-tube with 1 c.c. of pure sulphuric acid until no more iodine vapour is evolved. The iodine will condense on the sides of the tube, and, after the addition of a few drops of nitric acid, is converted into hydriodic acid by sodium bisulphite. Aristol is decomposed by heating 0.125 gramme with 2.5 c.c. of alcoholic potash (10 per cent.) and 0.5 gramme of powdered potassium nitrate, until the mass is dry and ignited. The residue is dissolved in water, reduced with sodium bisulphite and titrated as described above.

A. G. B.

On the Estimation of Sulphur in Petroleum. Fr. Heusler. (*Zeit. für angewand. Chemie*, 1895. Heft 10, pp. 285-286.)—Several of the methods for estimating sulphur in mineral oils (*e.g.*, those of Kast and Lagai. *Dingl.*, 284, 69, and Heusler, *Ber.*, 28, 493) cannot be relied upon when the amount to be determined is very small—0.02 gramme and less.

In his handbook of "Chemical Technology," Fischer describes a process for estimating sulphur in coal-gas, which consists in conducting the products of combustion through a tube bent many times, in which the sulphur is oxidized by a solution of hydrogen peroxide.

The principle of this method has been adapted by the author to the estimation of small quantities of sulphur in mineral oils. His apparatus consists of a small petroleum lamp, to be placed under a cylinder with an opening in the side near the bottom for air to enter, and an opening at the top for the gases to pass off. By means of a special contrivance, which cannot be clearly described without the illustration, the sulphur in the products of combustion is oxidized by a solution of potassium permanganate, which drops from a spiral worm, meets the gas, and then falls into a small flask.*

In making the estimation, 20 to 30 grammes of the oil are placed in the lamp, which is then weighed, lit, and placed under the cylinder. By means of an aspirator at the top a current of air is drawn through the apparatus, and is so regulated that the combustion is complete. The lamp may then be left burning for twelve hours or

* The complete apparatus may be obtained from C. Gerhardt, Marquart's Lager Chemischer Utensilien, Bonn.

longer. At the end of the experiment it is again weighed, and the weight deducted from the previous weight gives the amount of oil consumed. The permanganate solution is boiled with HCl and filtered, and the sulphuric acid precipitated with BaCl_2 .

C. A. M.

Estimation of Thiophen in Benzene. G. Deniges. (*Compt. rend.*, 1895, cxx., 781; through *Chem. Zeit.*)—This can be effected by precipitating the thiophen in the form of the compound $2(\text{HgO}, \text{HgSO}_4), \text{C}_4\text{H}_4\text{S}$, as follows: Two c.c. of the benzene are added to 20 c.c. of a solution of basic mercuric sulphate (50 grammes HgO , 200 c.c. H_2SO_4 , 1,000 c.c. water) contained in a well-stoppered flask (60 c.c.). The stopper is wired down, and the flask heated in a water-bath for about a quarter of an hour. When cold the liquid is filtered through a weighed filter, which is then washed with hot water, dried at $110-115^\circ$, and weighed. The increase in the weight of the filter is multiplied by the factor 0.0758 to obtain the weight of thiophen in the sample.

If objection be taken to the heating of the flask, this may be avoided by dissolving the benzene in some solvent miscible with water. A freshly-prepared mixture of 10 c.c. of the mercuric sulphate solution with 30 c.c. of acetone-free methyl-alcohol will dissolve 4 c.c. of pure benzene to a clear solution; but if thiophen be present a precipitate will form in a short time, and may be weighed in the manner described above. Care must be taken that the basic mercuric sulphate is in excess, otherwise the compound $2\text{HgO}, \text{HSO}_4, \text{C}_4\text{H}_4\text{S}$ is likely to be formed.

A. G. B.

Luteol; A New Indicator. W. Autenrieth (*Arch. Pharm.*, 1895, ccxxxiii., 43; through *Chem. Zeit.*)—Luteol or chlorohydroxydiphenyl-quinoline is obtained from phenacetin. It crystallizes in slender yellowish needles, melts at 246° , is insoluble in water and sparingly soluble in alcohol, and can be sublimed. It serves as an indicator in those cases where litmus or phenolphthalein is inapplicable. In acid liquids it is perfectly colourless, but intensely yellow in alkaline solutions.

A. G. B.

The Composition of some "Vegetable" Colouring Matters for use in Confectionery, etc. G. Posetto. (*Zeits. Nahrungsm. Unters. u. Hygiene*, 1895, ix., 150; through *Chem. Zeit. Rep.*, 1895, 188.)—A set of these "vegetable" colours, consisting of pastes mostly insoluble in water and alcohol, have been examined and were found to be tin lakes. When dry they contain from 72 to 80 per cent. of tin oxide, and as in their moist condition they contain about 70 per cent. of water, the amount of tin present must be at least 20 per cent. The blue paste is pure indigo-carmin, with 2.5 per cent. of ash, chiefly sulphates.

F. H. L.

A Characteristic Reaction of Citric Acid. L. Stahr. (*Nordisk pharm. Tidskrift*, 1895, ii., 141; through *Chem. Zeit. Rep.*, 1895, 187.)—If 10 mgrms. of citric acid be dissolved in 1 c.c. of water, a few drops of $\frac{N}{10}$ permanganate added and the

liquid warmed till the colour has vanished, and 3 to 5 drops of saturated bromine water dropped in, on cooling, a white crystalline precipitate appears, which, on the addition of caustic soda, yields the odour of bromoform. The reaction will detect 0.2 mg. of citric acid.

F. H. L.

The Estimation of 'Available' Phosphoric Acid in Thomas Slag. G. Sani. (*Staz. Sper. Ag. Ital.*, xxviii., 275.)—The author takes 5 grammes of the slag, adds 100 c.c. of water, and saturates at a boiling temperature with 10 per cent. citric acid solution, adds 200 c.c. of ammonium citrate solution; the mixture is then heated for an hour on the water-bath, with careful shaking, and made up to 500 c.c. The phosphoric acid dissolved is then estimated in the usual manner. H. D. R.

The Methods of Analysis of Copper Salts. L. Sostegni. (*Staz. Sper. Ag. Ital.*, xxviii., 167.)—The author shows that in the electrolytic method of determination of copper, arsenic, if present, is deposited at the same time. This method is too long, and requires too much attention.

Precipitation with zinc gave slightly high results, due to the deposition of a basic copper salt, and possibly, also, of cuprous chloride.

Titration with sodium sulphide, using as indicator paper prepared with alkaline lead acetate, was found to be inaccurate. The method of titration with ferrocyanide, using paper moistened with ferric chloride solution as indicator, was not found to be delicate enough for commercial purposes.

One gramme of the copper sulphate to be tested is dissolved in 25 c.c. of water and 15 c.c. of Soxhlet's alkaline tartrate solution added, and into this solution 20 c.c. of a 1 per cent. solution of glucose are dropped, little by little, the whole being boiled, and the boiling continued for five minutes. 25 c.c. of boiling water are now added, and after allowing the cuprous oxide to settle for a short time, the solution is decanted through a filter; boiling water is at once poured on the cuprous oxide, and the filter is then well washed before the precipitate is transferred to it. The cuprous oxide is then transferred to the filter, washed till the water which runs through is neutral, and the filter and its contents dried in a stove. The bulk of the precipitate is transferred to a platinum boat, the filter burnt and placed at one end of the boat, the whole then transferred to a tube, a piece of asbestos-paper being placed under the boat, and the Cu_2O reduced in a current of hydrogen. The results are very good, and iron does not interfere if present in traces. If notable quantities are present, the iron should be brought to the ferric state and precipitated by ammonia.

The cuprous oxide may be also converted into cuprous sulphide, and weighed as such, by placing a little sulphur in the boat (which should then be of porcelain) before placing it in the tube, and proceeding as before.

H. D. R.

Estimation of Tellurium in Copper Bullion. C. Whitehead. (*Jour. Amer. Chem. Soc.*, xvii., 1895, 280-281.)—From 25 to 50 grammes of the bullion are

dissolved in HNO_3 of 32° Bé, and the excess of acid removed by boiling. To this solution ferric nitrate solution, corresponding to 0.25 gramme of iron, is added. Then an excess of ammonia, and the precipitate is washed with dilute ammonia until all the copper salts are completely removed. If necessary, the precipitate is dissolved and re-precipitated. The residue on the filter, consisting of iron tellurite and selenite, is dissolved in HCl , an excess of tartaric acid added, the solution made alkaline with KOH , and H_2S passed through, when the selenium and tellurium dissolve as sulphides. The liquid is filtered, dilute HCl added to decompose the alkaline sulphides, and after standing in a warm place to remove H_2S , again filtered. The tellurium and selenium sulphides are dissolved in aqua regia, the solution evaporated to dryness, then taken up with HCl , and the tellurium and selenium precipitated by a current of SO_2 passed in to saturation. After standing twelve hours in a warm place, the precipitate is filtered on a weighed filter, dried at 100° , and the tellurium and selenium weighed together. The selenium is separated by boiling the precipitate in concentrated KCN solution, filtering, and adding HCl , when the selenium is deposited as a brick-red precipitate, which is dried at 100° and weighed.

The tellurium is estimated by difference, or the small amount dissolved by the KCN solution may be precipitated by SO_2 , after removal of the selenium, added to the original residue, and the whole dried at 100° and weighed.

C. A. M.

The Estimation of Antimony as Antimonious Antimoniate. Otto Brunck. (*Zeit. für anal. Chem.*, 1895, *Zweites Heft*, pp. 171-174.)—This method of estimating antimony has for some time past been in disrepute, since Bunsen showed that the tetroxide was volatile on ignition, and lost as much as 0.1 gramme in six hours. This observation being opposed to the general statements that Sb_2O_4 is infusible and non-volatile led the author to investigate the method.

Antimony, obtained pure by reducing potassium antimoniate with potassium cyanide, was weighed into a porcelain crucible and oxidized with HNO_3 , the crucible being covered with a watch-glass. The contents were then ignited in the crucible without a lid until constant in weight, and the amount of antimony calculated from the Sb_2O_4 found. In six determinations the amount found corresponded to within a milligramme of that taken. The uncovered crucible could be kept at a red heat for several hours without losing the slightest amount in weight.

Experiments were then made to discover under what conditions a loss by volatilization could occur, and it was found that the decomposition of the Sb_2O_4 into the trioxide and oxygen admitted of a simple explanation. Where the crucible was covered with a lid the flame gases were caught by its projecting edge, conducted into the crucible, and exercised a reducing action there. When the interior of the crucible had free contact with the atmospheric air, this reducing action was altogether obviated.

With the precaution of igniting the residue in an uncovered crucible the method is not only very rapid and exact, but where small quantities of antimony are to be estimated is to be preferred to any other.

C. A. M.

The Superiority of Barium Hydroxide Solution as an Absorbent in Carbon Determinations in Steel. J. O. Handy. (*Journ. Amer. Chem. Soc.*, 1895, xvii., pp. 247-251.)—The absorption of CO_2 by means of KOH in Geissler bulbs has the disadvantage that variations of the temperature and degree of moisture in the air of the laboratory produce errors which the greatest care can only partially eliminate. By using barium hydroxide instead of potash these errors are avoided, and it is possible to finish the analysis in at least three ways. 1. The barium carbonate may be filtered off, washed, ignited, and weighed. 2. The excess of barium hydroxide may be titrated (a) with standard oxalic acid without filtering; (b) with standard H_2SO_4 after filtering off the barium carbonate. Phenolphthalein is used as the indicator in each case, but the end reaction with H_2SO_4 is sharper.

In the estimation, 10 grammes or more of a steel containing 0.06 per cent. carbon, or 5 grammes of one containing 0.20 per cent., are dissolved by shaking in a stoppered bottle with a mixture of copper and potassium chloride solution (1 lb. in 1,400 c.c. of water) and HCl. 600 c.c. of the former and 40 c.c. of the acid are used for 10 grammes of steel, and proportionately more or less for other amounts. The liquid is filtered through asbestos which has been ignited in oxygen, and the carbon residue dried at 100° , and burned in the usual way in the combustion tube. The products of combustion pass through a U-tube with CaCl_2 in the first limb and anhydrous copper sulphate in the other, and then through two absorption tubes, each containing 50 c.c. of barium hydroxide solution. When the combustion is complete, and pure air or oxygen has been passed through to expel all carbon dioxide, the absorption tubes are disconnected and their contents filtered. During filtration air free from CO_2 is kept playing on the surface of the barium hydroxide solution, to prevent further formation of carbonate. The precipitate is washed with distilled water free from CO_2 , and the excess of barium hydroxide titrated with decinormal H_2SO_4 , of which 1 c.c. = 0.0006 gramme carbon. The barium hydroxide solution used contains about 20 grammes per litre.

The following results were obtained in experiments to test the method.

	KOH Method. Chemist "L," "C."	Ba(OH) ₂ Method. Chemist "H."	Colour Test. KOH Standard.
Steel "A" Carbon ...	0.14	0.134	0.14
" 11834.7 Carbon ...	0.109	—	0.11
" 11857.0 " ...	0.178	—	0.17
" "M" " ...	0.08	{ 0.090 0.091 and 0.093 0.092 }	0.08

C. A. M.

Lead Carbonate as an Agent for opening up Silicates. P. Jannasch. (*Zeits. Anorg. Chem.*, 1895, viii., 364; through *Chem. Zeit.*)—The silicate is fused with pure lead carbonate in a platinum crucible. The melt is loosened by plunging the hot crucible in cold water, and is dissolved in strong nitric acid. The silica is separated by evaporation to dryness, and the lead separated from the filtrate; on the addition of HCl, most of the lead separates as chloride, and the remainder is removed as sulphide. The lead carbonate is best prepared by precipitating lead acetate with ammonium carbonate, and washing with hot water.

A. G. B.

CORRESPONDENCE.

To the Editors of THE ANALYST.

The Yews, Reigate, July 11, 1895.

SIRS,—Your issue for this month contains the report of a paper read by Mr. W. C. Young, F.I.C., F.C.S., at the meeting of the Society of Public Analysts, on May 1 last, on "A Comparison of the Organic Carbon and Nitrogen Results obtained by Dr. Frankland and the Companies' Analysts from the Waters supplied by the Metropolitan Water Companies," and animadverting on my method of determining organic carbon and organic nitrogen.

Mr. Young does not seem to be aware that the cause of the discrepancies which he mentions had been already explained in my annual report to the Registrar-General for 1894, published in his "Annual Summary" for that year. As this "Annual Summary" is probably not extensively read by analytical chemists, I will thank you to insert in your next number the following extract, which has reference to the matter discussed in Mr. Young's paper:

"The relation between the amounts of organic carbon and organic nitrogen recorded in these tables affords data from which an opinion may be formed as to the origin of the organic matter, whether animal or vegetable. If the relative proportion of nitrogen to carbon be high, the inference is that the organic matter is chiefly animal; on the other hand, if it be low, it is certain that the organic matter is chiefly, if not entirely, of vegetable origin. Examined from this point of view, these tables indicate that the organic matter present in the river waters as delivered in London was, to a very large extent, of vegetable origin. In reference to this subject, the attention of the Local Government Board was recently called by the London County Council to the discrepancy between my results, as given in these tables in my former reports, and those furnished by the chemists who make analyses for the Metropolitan Water Companies, the results obtained by the companies' chemists indicating that the organic matter present in the waters was chiefly of animal origin, whilst, as just mentioned, my analytical results assigned to it chiefly a vegetable origin. At a consultation with these chemists in the month of June last it was discovered that, owing to an error in the method of analysis pursued by the companies' chemists, they had obtained results which showed too large a proportion of nitrogen to carbon in the organic matters. There had also been for a long time a considerable discrepancy between our analytical results in the case of the Chelsea Company's water, the cause of which was discovered at the same time. The assistant who collected the samples for the companies' chemists had, during two years, inadvertently taken samples of the New River Company's water, believing them to be drawn from the Chelsea Company's main. Consequently, the results reported as having been obtained by them with the Chelsea Company's water had, in fact, been got in the analyses of water drawn from the New River Company's mains. These errors were at once remedied, and since that time our analytical results have been in close accordance."

I will only add here, for the special information of analytical chemists, that the chief error in the method of analysis pursued by the companies' chemists consisted in the omission of an inch of oxide of copper in front of the copper gauze in the combustion tube. This omission causes the admixture of a small quantity of carbonic oxide with the gases resulting from the combustion; and, unless a special determination of carbonic oxide is made, this gas would of course be measured as nitrogen; thus increasing the amount of organic nitrogen and diminishing, to the same extent, that of organic carbon. The necessity for using the front layer of oxide of copper is fully described in my "Water Analysis" (1st edition, p. 66, and 2nd edition, p. 65); and a sketch, showing the different layers in the combustion tube, is given on a page immediately following. When the combustion process is conducted in accordance with the directions there given, it yields results which, as tested by duplicate analyses, are in at least as close agreement as any obtainable in other direct organic determinations.—I am, etc.,

E. FRANKLAND.

THE ANALYST.

SEPTEMBER, 1895.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

DUCLAUX'S METHOD FOR THE ESTIMATION OF "VOLATILE FATTY ACIDS," THE LAWS GOVERNING "VOLATILITY" DEDUCED THEREFROM, AND THEIR APPLICATION TO ANALYSIS, MORE ESPECIALLY TO THAT OF BUTTER.

By H. DROOP RICHMOND.

(Read at the Meeting, June 5, 1895.)

PART I.—THE MATHEMATICAL DEDUCTIONS FROM THE DISTILLATION OF ACIDS OF THE $C_nH_{2n+1}COOH$ SERIES.

DUCLAUX, from a study of the behaviour of the lower fatty acids of the series $C_nH_{2n+1}COOH$ on distillation in dilute aqueous solution, found that each acid had its own rate of distillation, whether distilled alone or mixed with its homologues, and used this as a means of estimating the members of this series. (*Ann. Chim. Phys.* [5] ii., 223.)

His experiments and method are well known, but the latter has not come largely into use; though Duclaux obtains excellent results with it, other experimenters are not successful in obtaining results comparable with his (*cf.* Allen, "Commercial Organic Analysis," i., 411; Violette, *Compt. Rend.*, cxi., 345; and Wilson, *Journ. Soc. Chem. Ind.*, ix., 18). I have from time to time used this method, and though my results are approximately in agreement with those of the originator, I have pointed out marked discrepancies (*Staz. Sper. Ag. Ital.* xxiii., 5); a recent study of the method has convinced me that Duclaux's method is vitiated by unsuitable conditions of experiment, and that his mathematical deductions from his results are erroneous.

Duclaux founds his theoretical considerations on the axiom that "the quantity of acid which vaporizes at a given moment is proportional to that which exists at the same moment in the heated liquid"; this mode of expression is unfortunate, as it is not definitely stated whether the quantity of acid existing in the liquid at a given moment is the absolute quantity, the quantity relative to the amount of liquid at that moment, or the quantity relative to the amount of liquid at the commencement of distillation; it is evident that the absolute quantity cannot be meant, and we are left with two alternatives. A little consideration shows that the quantity relative to the amount of liquid at the given moment is the correct mode of interpretation; whether 100 c.c. or 50 c.c., or any other quantity, of liquid is distilled, the vapour should have the same composition, and it is not reasonable to refer the composition of the liquid to some bulk which it had in the past. Duclaux, however, assumed the latter alternative, that the quantity of acid in the heated liquid is expressed in terms of the

bulk of liquid at the commencement; this is plainly shown by the form of equation deduced (*cf.* Wynter Blyth, "Foods," p. 450):

$$a = Y(1 - e^{-\frac{t}{T}}).$$

That this form of equation is not correct is shown by the fact that it either requires a proportion of acid distilled before the commencement of the distillation, or more or less than 100 per cent. when the distillation is complete—an assumption which it is difficult to make, and its fallacy is borne out by the equation agreeing only with a portion of the results.

I have studied the distillation of butyric acid (*loc. cit.*), and proposed an equation of the form

$$x = Ay - By^2 + Cy^3.$$

In this I fell into an error in the mathematical deductions, *i.e.*, that both the quantity of acid distilled and the condensation in the retort were functions of the volume of the distillate, and a further study has now revealed the error.

Starting from Duclaux's axiom, it follows that the relation between the composition of the vapour given off at any moment and the composition of the liquid should be constant; this is Henry's law of the distribution of a gas between a space and a solvent. In other words, the volatile fatty acids in dilute solution may be assumed to exist as perfect gases, which is in accordance with recent theories of solution. The relation between the composition of the vapour and the composition of the liquid should be the inverse ratio of the vapour-pressure of the acid $C_nH_{2n+1}COOH$ as a perfect gas at $100^\circ C.$ to the solubility of the acid in water at $100^\circ C.$; or, in other words, the rate of distillation is an inverse function of the solubility of the acid in water.

The apparent paradox pointed out by Duclaux, that the higher the molecular weight of the acid, and also boiling-point, the faster is the rate of distillation, is easily explained. For example, butyric acid boils at $162.5^\circ C.$, but when a dilute aqueous solution is distilled, it passes over faster than water. We in this case measure the boiling-point of a compound $(C_3H_7COOH)_n$, but in dilute aqueous solution the compound C_3H_7COOH exists, the acids $C_nH_{2n+1}COOH$ being unknown in the pure state (*cf.* Ramsay and Shield, *Journ. Chem. Soc.*, lxxviii, 1089).

The general equation that I have deduced from the results obtained by Duclaux (and in the case of butyric acid by Wollny and myself) is

$$(100 - y) = \frac{(100 - x)^a}{100^{a-1}} \times K^{-a}.$$

x = percentage volume distilled.

y = percentage of acid distilled.

The expression K^{-a} is, as I shall show, an approximation to the condensation in the retort, and may be neglected here. The expression then stands

$$100 - y = \frac{(100 - x)^a}{100^{a-1}} \dots (1).$$

The quantity of acid existing in the retort at any moment is

$$\frac{100 - y}{100 - x} = \frac{(100 - x)^a}{100^{a-1}} = \frac{(100 - x)^{a-1}}{100^{a-1}} \dots (2).$$

The quantity of acid vaporizing at that moment is the first differential coefficient of

$$(1) = a \frac{(100 - x)^{a-1}}{100^{a-1}} \dots (3).$$

The ratio of the composition of the vapour to that of the liquid is the ratio of (2) to (3), that is, a , which shows that equation (1) fulfils Duclaux's axiom, and also Henry's law. The effect of condensation in the retort is of importance, as it follows from Henry's law that the composition of the condensed liquid will not be the same as that of the vapour; if a is greater than 1, the condensed liquid will be weaker than the vapour, and if less, stronger; if a is greater than 1, the condensed liquid will also be stronger than the liquid in the retort, and if a is less, weaker. The effect of this being cumulative, a considerable deviation from equation (1) may be noticed. The loss by condensation is of course a function of the composition of the vapour, and I have expressed it as a logarithmic function of the volume of the distillate. Owing to the condensation being small, the composition of the vapour may be assumed, without great error, to bear an approximately constant proportion to its bulk, and my expression K^{-x} , though not really correct, is a sufficiently close approximation; of course, this correction is only applicable if the perfect gaseous state is assumed, and where it is not may disappear, and the value of the sign may even be reversed.

There is also a loss by imperfect condensation, unless special precautions are taken, and when a exceeds 1, this loss may make a noticeable difference; thus I have found a loss of 2 per cent. of the total amount of butyric acid on distilling a dilute solution, when the total loss of liquid by imperfect condensation has been 0.4 per cent. I have assumed that this loss is directly proportional to the acid distilled, and have eliminated the source of error by taking the total amount of acid to be the sum of the distillate, and that remaining in the retort and condenser; I have corrected some of Duclaux's series on this account.

The corrections on account of condensation being small, the error of these will lie within the limits of experimental error.

The following values of a and K in the general equation

$$100 - y = \frac{(100 - x)^a}{100^{a-1}} \times K^{-x}.$$

	a .	K .
Formic acid (Duclaux)	0.4	1.00079
Acetic	0.667	1.
Propionic acid (Duclaux)	1.111	1.000723
Butyric	2	1.
(Wollny) ...	2	1. (?)*
(Richmond)	2	1.0023451
Valeric (Duclaux) ...	3	1. (?)
Caproic	4	1.003508
Caprylic	8 (?)	(?)

In the following tables are given the experimental results and the calculated values. Column I. contains the percentage volume of the distillate; Column II. the percentage of acid distilled; Column III. the corrected values of this (when neces-

* K in this case is probably not constant, as W. used a 5 per cent. solution, and it is probable that the perfect gaseous state was not attained.

sary); Column III. the calculated values of y ; Column IV. the difference; Column V. the strength of the liquid in the retort; Column VI. the strength of the vapour given off; and Column VII. the ratio between them.

FORMIC ACID.

I. x	II. y	III. y (calc.)	IV. diff.	V.	VI.	VII. a
9.09	3.5	3.5	—	106.2	41.2	.39
18.18	7.6	7.3	-.3	113.3	45.1	.40
27.27	11.8	11.5	-.3	121.3	47.3	.39
36.36	16.3	16.1	-.2	131.5	52.8	.40
45.45	21.6	21.1	-.5	143.7	60.5	.42
54.54	27.3	26.8	-.5	160.0	66.0	.41
63.63	33.7	33.3	-.4	182.3	77.0	.42
72.72	41.4	41.0	-.4	214.9	92.7	.43
81.81	50.7	50.5	-.2	271.4	118.5	.44
90.9	63.5	63.5	—	401.5	208.5	.5

ACETIC ACID.

I.	II.	III.	IV.	V.	VI.	VII.
9.09	5.9	6.2	+ .3	103.5	67.1	.65
18.18	12.2	12.5	+ .3	107.3	70.5	.66
27.27	18.8	19.1	+ .3	111.7	73.7	.66
36.36	25.6	26.0	+ .4	117.0	77.2	.66
45.45	32.7	33.3	+ .4	123.1	81.5	.66
54.54	40.5	40.8	+ .3	131.6	86.7	.66
63.63	48.7	49.0	+ .3	140.9	95.2	.67
72.72	57.9	57.9	—	154.2	104.5	.68
81.81	67.7	67.9	+ .2	177.5	119.3	.67
90.90	79.8	79.8	—	222.2	166.5	.75

PROPIONIC ACID.

I.	II.	III.	IV.	V.	VI.	VII.
9.09	10.5	10.6	+ .1	98.5	116.0	1.18
18.18	21.1	21.0	-.1	96.4	115.5	1.20
27.27	31.5	31.2	-.3	94.2	111.6	1.18
36.36	41.4	41.1	-.3	92.2	108.9	1.18
45.45	51.3	50.7	-.6	89.0	105.6	1.18
54.54	60.6	59.9	-.7	86.6	100.1	1.16
63.63	69.5	68.9	-.6	86.5	95.7	1.11
72.72	78.0	77.6	-.4	80.6	90.0	1.12
81.81	85.8	85.8	—	77.8	81.4	1.05
90.90	92.8	93.5	-.7	79.0	79.1	1.0

BUTYRIC ACID (WOLLNY).

I.	II.	III.	IV.	V.	VI.	VII.
10	18.2	19.0	+ .8	90.9	176.6	1.94
20.	35.3	36.0	+ .7	80.9	161.7	2.00
30	50.4	51.0	+ .6	70.9	140.3	1.98
40	63.6	64.0	+ .4	60.7	121.0	1.99
50	74.9	75.0	+ .1	50.2	104.0	2.07
60	84.2	84.0	-.2	39.5	82.5	2.09
70	91.1	91.0	-.1	29.7	62.0	2.09
80	96.3	96.0	-.3	18.5	40.0	2.16

BUTYRIC ACID (DUCLAUX).

I.	II.	IIa.	III.	IV.	V.	VI.	VII.
9.09	17.1	17.4	17.4	—	90.9	184.2	2.03
18.18	32.7	33.4	33.1	- .3	81.4	164.1	2.02
27.27	46.3	47.1	47.1	—	72.7	144.1	1.98
36.36	58.5	59.5	59.5	—	63.6	126.5	1.99
45.45	68.8	70.0	70.3	+ .3	55.0	106.7	1.94
54.54	77.5	78.8	79.3	+ .5	46.6	88.0	1.89
63.63	84.3	85.9	86.7	+ .8	38.8	72.6	1.87
72.72	90.2	91.8	92.5	+ .7	30.7	58.3	1.90
81.81	94.6	96.3	96.7	+ .4	20.4	43.3	2.12
90.90	97.5	99.2	99.2	—	8.9	28.6	3.2

BUTYRIC ACID (RICHMOND).

I.	II.	III.	IV.	V.	VI.	VII.
9.09	19.1	19.1	—	89.0	195.9	2.20
18.18	35.4	35.1	- .3	78.6	170.5	2.18
27.36	50.0	50.5	+ .5	68.8	150.4	2.18
36.45	62.6	62.8	+ .2	58.9	127.6	2.17
45.73	73.1	73.1	—	49.6	103.6	2.09
54.82	81.3	81.5	+ .2	41.4	85.0	2.05
64.09	88.5	88.5	—	32.0	70.2	2.19
73.18	93.7	93.7	—	23.5	50.0	2.13
81.90	97.4	97.2	- .2	14.3	34.1	2.4
91.45	99.3	99.4	+ .1	8.2	17.6	2.15

VALERIC ACID.

I.	II.	IIa.	III.	IV.	V.	VI.	VII.
9.09	24.5	25.2	24.9	- .3	82.3	254.6	3.09
18.18	44.5	45.8	45.3	- .5	66.2	202.4	3.06
27.27	59.5	61.3	61.6	+ .3	53.2	152.9	2.87
36.36	71.3	73.2	74.3	+ 1.1	42.1	111.4	2.65
45.45	79.5	81.9	83.8	+ 1.9	33.2	84.7	2.6
54.54	85.7	88.3	90.6	+ 2.3	25.7	59.4	2.3
63.63	89.7	92.4	95.2	+ 2.8	20.9	42.1	2.0
72.72	92.8	95.6	98.0	+ 2.4	16.1	33.0	2.1
81.81	95.4	98.3	99.4	+ 1.1	9.3	25.0	2.7
90.90	96.9	99.9	99.9	—	?	?	?

CAPROIC ACID.

I.	II.	III.	IV.	V.	VI.	VII.
9.09	33.5	33.9	+ .4	73.2	320.1	4.4
18.18	56.0	58.0	+ 2.0	53.8	232.2	4.3
27.27	75.5	74.5	- 1.0	38.7	179.9	5.3
36.36	86.0	85.6	- .4	21.7	98.0	4.5
45.45	92.5	92.5	—	13.7	61.0	4.5
54.54	96.5	96.5	—	7.7	37.4	4.9
63.63	97.5	98.6	+ 1.1	6.8	10.5	1.5
72.72	98.4	99.6	+ 1.2	5.9	9.9	1.7
81.81	99.3	99.9	+ .6	8.8	8.8	2.8
90.90	100.0	100.0	—	?	?	?

Duclaux's figures for caprylic acid do not agree very well with a formula of the type given above, but the approximate value of a is 8.

The agreement, except in the case of valeric acid, is, considering the nature of the experimental error, good. The three series for butyric acid are interesting, as Wollny used a 5 per cent. solution, Duclaux a 1 per cent. solution, while mine was of about 0.3 per cent. strength, and though the different effect of condensation is plainly shown, yet our results all lead to the same value for the rate of distillation.

The figures in Column VI. for the strength of the vapour are obtained by interpolation, and from the quantities distilled; they do not, however, strictly represent the composition of the vapour in the retort, as not only is the condensation in the retort affecting this result, but a small error will be introduced during the condensation in the condenser, the condensed liquid not having exactly the same composition as the vapour; after allowing for this, and for the error in calculating the figures in Column VI., we see that the figures in Column VII. are approximately constant, and are nearly of the value of a in the formula.

It is evident that though a is constant for each acid, K will depend on the conditions of work, and this explains why other experimenters do not obtain results comparable with those of Duclaux. I am engaged in making experiments with an apparatus modelled on that of Brown (*Journ. Chem. Soc.*, xxxix. 304), and thus hope to eliminate the function K^{-a} .

The following laws of the volatility of the acids $C_nH_{2n+1}COOH$ may be laid down:

- (i.) Each acid of the series $C_nH_{2n+1}COOH$ on distillation in dilute solution behaves as a perfect gas and conforms to Henry's law.
- (ii.) Each acid has a fixed rate of distillation, which is an inverse function of its solubility in water, and is quite independent of the properties of the pure acids.
- (iii.) The apparent rate of distillation may be modified by condensation in the retort.

(To be continued.)

Ang-Khak, a Chinese Dye used for the Coloration of Articles of Food, etc.
H. C. Prinsen. (*Chem. Zeit.*, 1895, xix., 1311.)—Ang-khak is a purple-coloured substance formed by the action of a particular ferment on cooked rice, but the exact method of its preparation is unknown. In order to detect its presence in red wine, the liquid should be shaken with chloroform; if the latter remain colourless, ang-khak is absent. In the event of any red colour being extracted, a large quantity of the wine should be evaporated to dryness with sand, extracted with chloroform, and examined. It may be distinguished from any of the aniline colours by being precipitated by mercuric oxide, and by some other colour-reactions, which are indicated in the table. A is a pure red wine, C a 10 per cent. solution of ang-khak in alcohol, and B a mixture of four parts of wine and one of C.

	A.	B.	C.
1 c.c. wine + 4 c.c. soda (1 : 200) ...	Greenish.	Greenish-brown.	Red.
1 c.c. wine + 3 c.c. NH_3 (1 : 100) + 5 c.c. water ...	"	Brown.	Red.
4 c.c. wine + 1 c.c. alum (1 : 10) { filtrate	Colourless.	Red.	"
+ 1 c.c. soda (1 : 10) filtered { ppt.	Gray.	Violet.	"
2 c.c. wine + 1 c.c. lead acetate { filtrate	Colourless.	Red.	"
{ ppt. ...	Gray.	Violet.	"
10 c.c. wine + 0.5 grm. HgO —filtrate ...	Colourless.	Colourless.	Colourless.
5 c.c. wine + 2 c.c. chloroform, the chloroform is ...	Colourless.	Red.	Red.

F. H. L.

Some Samples of Italian Butter. P. Spallanzani and A. Pizzi. (*Staz. Sper. Ag. Ital.*, xxxviii., 257.)—Fortnightly analyses of butters from six localities in the province of Reggio, and of a few from the provinces of Modena and Parma, were made for the twelve months, March, 1894, to February, 1895. The Reichert-Wollny method was used without modification for the determination of the volatile acids, and the density of most of the samples and the refractive index of a few were also determined. One of the six localities chosen was the dairy attached to the Dairy School of Reggio, and the butters were prepared in the other five localities under the continued supervision of trustworthy and competent persons, and came from dairies where the care and good faith of the proprietors were considered above suspicion.

The lowest Reichert-Wollny figure obtained was 19.80 c.c., and the highest 30.14 c.c.; the density at 100° C. varied from 0.8640 to 0.8680, and the refractive index, measured with Zeiss' butyro-refractometer at 35° C., from 47.2 to 43.5.

The authors note that of the butters manufactured at the Dairy School at Reggio but two were below 25 c.c. (viz., 24.64 and 24.53), while in the other five localities butters giving below this figure were almost universal in November, December, January, and February. In all parts it was found that during the months of May, June, July, and August the Reichert-Wollny figure was uniformly high. The butters which gave low Reichert-Wollny figures were obtained when the yield was very small, and consequently, although the numeric mean was found to be 25.84 c.c., the average taking quantities produced into account was over 28 c.c., a normal figure.

The authors discuss the influence of feeding, and show that when the cows are out to grass the butter is rich in volatile acids, and when they are stall-fed, and on a poor ration, the Reichert-Wollny figure is low. They also show that higher figures are yielded in the early stages of lactation than in the later stages. A newly-calved cow yielded butter of Reichert-Wollny 28.05, while one which was at the end of its period of lactation yielded butter of Reichert-Wollny 21.23 c.c., the breed, feeding, and other conditions being the same in both cases.

They are of opinion that the larger globules of fat in milk are richer than the smaller ones, though they offer no experimental evidence of this (for an experiment showing that the large and small globules have the same composition, see ANALYST, xix. 76), and from this conclude that the mode of butter-making adopted may have an influence on the composition.

They conclude that (i.) butters with a greater refractive index at 35° than 48°, and a lower density at 100° than 0.8640, can be declared adulterated.

(ii.) A Reichert-Wollny figure of less than 20 c.c. shows the presence of foreign fat; figures higher than 20 and less than 26 c.c. in May to September, and less than 23 c.c. in the other months, are suspicious (in Emilia), and appeal to the cow should be resorted to.

H. D. R.

The Detection of Watered Milk by the Examination of the Milk Serum.

Dr. Lesceur. (*Congres Intern. de Chim. Appliq., Comp. Rend., Brussels, 1894*, pp. 60-62.)—A means of distinguishing between a watered milk and one from which cream has been abstracted is furnished by examination of the milk serum. Coagulation of the milk is readily brought about by adding a trace of rennet, and the serum is separated by filtration.

The density of milk serum thus obtained varies from 1.029 to 1.031 at 15°. In certain samples it has been found as low as 1.027, and this the author takes as his minimum limit.

The extract of the serum should also be determined. The weight varies from 67 to 71 grammes per litre, the mean being 70 grammes and the minimum 67 grammes.

Every sample therefore which yields a serum with a density lower than 1.027, and the extract of which does not reach 67 grammes per litre, should be looked upon as watered.

The following figures show the effect of added water on the serum of a pure milk:

			Density of Serum at 15°.		Proportion of Extract per litre of Serum.
Pure milk	1.030	...	70 grammes
"	+ 10 % water	...	1.0275	...	64 "
"	+ 20 "	...	1.0251	...	59 "
"	+ 30 "	...	1.023	...	54.5 "

From this it appears that the addition of about 4 per cent. of water lowers the specific gravity by one-thousandth and the weight of the extract by two units.

In milk which has curdled naturally, the serum, in spite of its different composition, gives almost the same results as does the neutral serum prepared by rennet. Hence no modification of the method is necessary for curdled milk.

C. A. M.

The Chemical Variations of Different Kinds of Mace. **E. Späth.** (*Forschungsber. Lebensmittel. Hyg., etc., 1895, ii., 148; through Chem. Zeit. Rep., 1895, 202.*)—Banda mace may be distinguished from its usual adulterant, Bombay mace, by its containing about ten times as much essential oil. On the other hand, the latter contains more (56 per cent.) fat than the former (21 per cent.). The author has extracted this fat from a number of samples of mace from different sources by means of petroleum spirit and determined its constants. The figures obtained from mace from Banda, Menado, Penang, Macassar, and Zanzibar closely

agree with one another, being as follows: Melting-point in open tube, 25° to 26°; saponification number, 169.9 to 173; iodine number, 75.6 to 80.8; Meissl's number (for Banda only), 4.1 to 4.2; value in Zeiss's refractometer at 40°, 76 to 85; and coefficient of refraction, 1.480 to 1.487. Bombay mace gives the following: Melting-point, 31° to 31.5°; saponification number, 189.4 to 191.4; iodine number, 50.4 to 53.5; Meissl's number, 1.0 to 1.1; Zeiss, 48 to 49; coefficient of refraction, 1.463 to 1.464. The fat of mace "scales, i.e., the covering inside the seed mantle," differs considerably from that of the arillus itself, especially in melting-point (28.5° to 29°) and in saponification number (148.2 to 148.8); its iodine number is also low (71.3 to 73.4).

F. H. L.

On the Isolation, Quantitative Separation, and Chemical Characteristics of Alkaloids and Glucosidal Bodies in Forensic Cases. Carl Kippenberger. (*Zeit. f. anal. Chem.*, 1895 [3], pp. 294-346.)—The investigations of the author have been made with special reference to the detection of poisonous alkaloids in the dead body. The products of the decomposition of animal cells after death, which in certain cases give with tannic acid, iodine, and other reagents reactions simulating those of alkaloids, have hitherto been among the chief difficulties in the examination. While many have studied the reactions of these cadaveric "alkaloids," but little work has been done to determine their chemical constitution, and in isolating them too little care has been taken to ensure that albumin and its further decomposition products have been absent. In the author's opinion, which is based on experiment, the reactions obtained with the so-called cadaveric alkaloids are in many cases due to the presence of albuminoid compounds, and there is good reason to doubt whether among the normal decomposition products of the dead body any substances occur which are completely analogous in chemical composition to the vegetable alkaloids.

It is of the greatest importance, therefore, to remove peptone and albumin from the extract, and this may be most readily done by precipitation with tannic acid, no trace of them being then found in the filtrate. Most alkaloids are also precipitated with tannic acid, but these alkaloidal compounds are soluble in fluids containing glycerin, and may thus be separated in a pure state. The author's experiments on extracting parts of the decomposed body with and without the addition of vegetable alkaloids completely confirm the value of this means of separation. The action of the glycerin on the compounds of the alkaloids and tannic acid is not merely that of a solvent, for it is shown that a definite compound is formed with them.

For removing the alkaloids from the extracted fluid, chloroform gives better results than ether as used in the Stas-Otto method. It does not take up water from the aqueous solution, is a much better solvent for many glucosidal bodies, and gives a more quantitative removal, especially with strychnine and atropine. The glycerin separates well from the chloroform when the latter is present in at least equal volume. The best solvent for removing fat is petroleum spirit with a boiling-point of from 30° to 35° C. It separates readily, and does not dissolve in the glycerin.

The alkaloids, morphine, narceine, curarine, and the poisonous substance strophantin, will not go into solution in chloroform, ether, etc., either from acid or alkaline (fixed alkalies) solution. With the exception of curarine they dissolve, how-

ever, when shaken up with ammonia solution, with the addition of a little warm amyl alcohol. The drawback to this is the impurities contained in the amyl alcohol.

Saturation of the solution with common salt offers a means of separating many alkaloids from aqueous solution, and they may then be taken up by a solvent which does not mix with the salt solution.

Further experiments showed that morphine is precipitated by alkaline carbonates, and that narceine, while not so insoluble, is still rendered capable of being shaken out with chloroform containing some alcohol. If, then, in the second stage of shaking out, alkaline bicarbonate be added to the liquid made alkaline with alkaline hydroxide, morphine and narceine are removed by shaking with chloroform containing 10 per cent. of alcohol. The alkaloids may be recovered from the separated chloroform by shaking with acidified water.

Strophantin remains most stubbornly in the aqueous solution, but may be separated by means of a mixture of equal parts of chloroform and ether, in which it dissolves after being shaken both in acid and alkaline solution.

The general method of procedure used by the author is as follows :

1. The animal matter to be examined for poisons is extracted for two days at about 40° C., with glycerin containing a large amount of tannic acid in solution, with or without the addition of water. The fluid, after the solid matter has been removed, is freed from blood fibrin and dissolved albumin* by heating to about 50° C. and filtering after cooling.

- * 2. Fat is removed by shaking up twice with petroleum spirit, the last traces of the latter being removed by heating on the water-bath. The alkaloids are then shaken out with chloroform (a) from acid solution, (b) from alkaline solution, (c) from alkaline bicarbonate solution. Finally, the fluid is shaken with a mixture of chloroform and ether after saturation with common salt.

The treatment with petroleum spirit removes traces of veratroidine and jervine, besides the fat. On shaking the acidified liquid with chloroform, the following alkaloids are removed: Colchicine, digitaline, picrotoxine, cantharidine, papaverine, aconitine, narcotine, jervine, geissospermine, small quantities of delphinine, brucine, and veratrine, with traces of narceine and strychnine.

The fluid made alkaline with alkaline hydroxide and shaken with chloroform yields atropine, codeine, emetine, brucine, veratrine, sparteine, coniine, nicotine, strychnine, pilocarpine, apomorphine, and any narcotine and papaverine left from the previous shaking. Chloroform removes from the solution, after the addition of alkaline bicarbonate, morphine and narceine, while strophantin is extracted as described above.

Instead of direct extraction with the glycerin tannic acid solution, the extract may be first obtained by the usual Stas-Otto method, and then subjected to the method of treatment proposed by the author. This has, however, the disadvantage that certain inorganic poisons if present dissolve in the glycerin, and a more tedious process will be necessary to detect them.

* "vom Blutfibrin und eventuell gelösten Albumin durch Erhitzen auf circa 50° C. befreit."

ESTIMATION, SEPARATION, AND CHEMICAL CHARACTERISTICS OF ALKALOIDS AND GLUCOSIDE BODIES.

A. Methods for the Estimation of Single Alkaloids.

The estimation of an alkaloid when present in the pure state is readily carried out by controlling the gravimetric results by titration with acid. In forensic cases this cannot as a rule be done, owing to the chloroform taking up traces of acid and alkali during the shaking out. The gravimetric determination must therefore be controlled by a titration method applicable to acid and neutral solutions of the alkaloid. For this purpose solutions of iodine or mercuric iodide in potassium iodide are suitable; also, in special cases, a solution of mercuric chloride.

Iodine Solution.—R. Wagner (*Journ.*, 1861, p. 867) proposed precipitating the alkaloid in solution with excess of iodine, and titrating back with thiosulphate. Schweissinger (*Arch. d. Pharm.*, 1885, 615) showed that the presence of mineral acids did not affect the result, but that the alkaloid compound gradually decomposed on contact with water, and to obviate this he recommended filtering rapidly under pressure and titrating the excess of iodine in the filtrate. The author proves that this method, if carried out under certain precautions, gives results corresponding closely to those required by theory: A measured volume of the alkaloid solution is placed in a glass-stoppered flask and diluted with a measured volume of water. Iodine solution ($\frac{1}{10}$ N) is then added till no further precipitate forms, and the liquid well shaken. After standing from five to ten or fifteen minutes, according to the character of the alkaloid, it is filtered through asbestos, and the excess of iodine determined with $\frac{1}{10}$ N thiosulphate solution in an aliquot part of the filtrate. All the alkaloids tried gave good results with the exception of brucine. This is explained by the author on the supposition that the brucine compound is more readily decomposed by water, and that, in addition to the compound Alk. HI.I_2 of the alkaloids in general, the compound Brn. HI.I is also formed.

In titrating morphine salts, the presence of acid (HCl) and of NH_4Cl exercises a solvent action. With the other alkaloids they do not interfere, but rather promote the separation.

As a general rule, titration of the alkaloids with iodine solution is to be recommended except in the case of brucine.

Mercuric Iodide in Potassium Iodide Solution (Mayer's Reagent).—This gives with most alkaline solutions an insoluble precipitate, on which fact a titration method is based. The solution contains 13.546 grammes HgCl_2 and 49.8 grammes KI per litre. The 13.546 grammes HgCl_2 correspond to 12.69 grammes I , from which the amount of alkaloid is calculated. The method gives useful results, but too large an excess of KI must be avoided. The author points out that 1 c.c. of the solution does not correspond to 0.0213 gramme of narcotine, as given in the text-books, but to 0.02065 gramme.

Mercuric Chloride Solution.—The author has experimented with mercuric chloride solution, which in many cases gives an insoluble precipitate with alkaloids. Although in certain cases good results may be obtained, the number of alkaloids

with which the method can be used is limited, and there is no superiority over titration with iodine solution.

B. Separation of Mixtures of Alkaloids, and Chemical Characteristics of Individual Alkaloids and Glucosides.

1. *Alkaloids and Glucosides removed by Chloroform from Acid Solution.*—From the acidified tannic acid solution there separate the tannin compounds of colchicine, digitalin, papaverine, narcotine, delphinine, aconitine and agaricine, while picrotoxin and cantharidin remain in solution.

Of these the following are soluble in water made alkaline with alkaline hydroxide: Cantharidin and picrotoxin, agaricine, colchicine and digitaline, while delphinine, aconitine, narcotine and papaverine are insoluble.

By "acidified tannic acid solution" the author means his separation-reagent described above, to which HCl or H_2SO_4 has been added to turbidity, and then water until it just becomes clear again.

Separation of Aconitine, Narcotine, and Papaverine.—The picrates are formed by decomposing the alkaloid salts with an aqueous solution of picric acid. While wet the insoluble picrates are washed with ammonia solution. Narcotine and papaverine dissolve, while aconitine is left. The solution must not be too dilute since aconitine picrate is slightly soluble in water. With rapid filtration accurate results may be obtained.

Separation of Narcotine and Papaverine.—This is best effected in the form of oxalates. The di-oxalate of papaverine is almost insoluble in cold alcohol and in water, while the corresponding compound of narcotine is readily soluble.

Potassium ferricyanide precipitates papaverine from its neutral solutions. After standing twenty-four hours the separation is quantitative. The analogous compound of narcotine is difficultly soluble, but the separation is not quantitative. With delphinine and aconitine there is no precipitation.

Potassium ferrocyanide gives a precipitate with papaverine, and a similar one with narcotine, but in neither case is it quantitative. With delphinine it forms a blue precipitate.

With sodium nitroprusside papaverine forms a compound somewhat soluble in pure water and readily so in HCl. The compound with narcotine is almost insoluble in pure water, but is fairly soluble in acidified water, and readily soluble in alcohol and acetone. With delphinine the reagent forms a white precipitate, which becomes red on treatment with concentrated H_2SO_4 , and dissolves in excess of nitric acid, forming a yellow solution.

Colchicine.—A characteristic reaction is the orange colour which appears on adding hydroxylamine hydrochloride followed by a slight excess of NaOH. Strong HNO_3 gives a brown colour with the alkaloid, especially on warming; on diluting and adding NaOH, this becomes orange red.

Delphinine.—This has only the reaction with tannic acid in common with digitalin, while it differs from picrotoxin in its entire behaviour. It may be readily separated from the latter by treatment with alkaline hydroxide, in which picrotoxin is readily soluble. With concentrated H_2SO_4 delphinine gives a brownish solution

resembling that given with digitalin. The reaction with potassium ferrocyanide is also very distinctive.

Cantharidin is not an alkaloid, but an acid anhydride. It is more soluble in chloroform than in ether and benzene. No new reactions are given.

Papaverine.—A most characteristic reaction is the behaviour of its chromic acid compound with H_2SO_4 , a solution being formed in which, on shaking, violet stripes momentarily appear. Potassium bichromate gives a reddish-yellow precipitate with papaverine solutions. On adding H_2SO_4 to this on a tile, a green solution is formed, which gradually becomes brown.

Aconitine.—No alteration appears in the solution on adding potassium ferrocyanide, monochromates or bichromates, copper sulphate, Fehling's solution or lead acetate.

Agaricine.—A yellow powder, insoluble in acids, easily soluble in alkaline hydroxide. The tannic acid compound is readily soluble in excess of tannic acid. It slightly reduces Fehling's solution, but gives no characteristic reactions with H_2SO_4 , HNO_3 , or chromic acid compounds.

2. *Alkaloids removed by Chloroform from Solutions made Alkaline with Alkaline Hydroxide*.—The tannin compounds of the following are insoluble in acidified tannic acid solution: Brucine, strychnine, emetine, veratrine, narcotine, codeine and thebaine.

The analogous compounds of atropine, sparteine and nicotine are soluble.

Further Separations.—Any aconitine, papaverine, or narcotine still left in the solution can be removed by acidifying and shaking out with chloroform.

Nicotine, coniine and sparteine are liquid and volatile, and may be separated from the other alkaloids, including atropine, by distillation.

Brucine and Strychnine.—The most suitable method of separation is based on that of Dunstan and Short,* which depends on the fact that potassium ferrocyanide precipitates strychnine from acid solution, while the analogous brucine compound is soluble. The precipitate is decomposed with ammonia, the base removed by chloroform, dried at 105° , and weighed.

This was modified by Beckurts and Holst,† who added a standard ferrocyanide solution until all the strychnine was precipitated, iron chloride paper being used as the indicator.

The author has obtained accurate results by adding the ferrocyanide in slight excess, as shown by an indicator, allowing to stand for fifteen minutes, washing the precipitate as rapidly as possible, decomposing with ammonia, shaking out with CHCl_3 , and titrating the solution with $\frac{1}{20}$ N iodine solution.

Gerock's‡ method of separating brucine and strychnine as picrates also gives useful results, but is not so good as the ferrocyanide method. Since no insoluble compound with potassium ferrocyanide is formed by atropine, codeine, emetine, veratrine, narcotine or sparteine, this reagent can be used for the separation of strychnine from these alkaloids.

Separation of Brucine from Veratrine, Codeine, and Emetine.—Otto§ gives a

* *Pharm. Journ. and Trans.*, iii., 694.

† *Pharm. Centralhalle*, 1887, p. 119.

‡ *Arch. d. Pharm.*, 1889, p. 159.

§ *Anleit. zur Au-mitt. der Gifte*, 6 Edit., p. 67.

method for separating strychnine and brucine which depends on the solubility of the chromic acid compound of brucine in dilute acetic acid of sufficient concentration. But the author finds that the brucine compound is deposited after standing twenty-four hours. It is a yellow crystalline substance, and, according to the analysis, has the formula $(C_{28}H_{20}N_2O_4)_2K_2CrO_4$. On this fact he has based a separation method.

The alkaloids are dissolved in as little water as possible, neutralized with alkaline hydroxide, and the solution made feebly acid with acetic acid. A definite volume of H_2CrO_4 solution is added (9.725 grammes per litre), the liquid well shaken, and allowed to stand in a stoppered flask for twenty-four to thirty-six hours. The excess of chromate is then determined in an aliquot part of the filtrate by adding KI and HCl in excess and rapidly titrating the liberated iodine before the somewhat insoluble compounds of iodine with veratrine, codeine, or emetine have been formed.

The brucine chromate may also be estimated gravimetrically by washing with alcohol and ether and drying until the weight is constant.

With potassium bichromate brucine gives a dark-yellow precipitate, which is practically insoluble in dilute acetic acid. Veratrine only gives a precipitate in concentrated solution, and this gradually dissolves on adding excess of acetic acid. With codeine, emetine, and atropine there is no precipitate with bichromate, either in neutral or acid solution.

Brucine and Thebaine.—These can be separated by precipitation with bichromate from strongly acid solution, brucine bichromate being but little soluble under these conditions.

Brucine, Veratrine, and Thebaine.—The alkaloids are dissolved in alcohol, and a solution of mercuric chloride in alcohol added. After standing from twenty-four to thirty-six hours, the brucine compound is precipitated, while those of veratrine and thebaine remain in solution.

The behaviour of the picrates of these alkaloids towards ammonia solution is another means of separation. Brucine picrate is insoluble in ammonia solution, veratrine picrate somewhat soluble, while strychnine picrate is fairly soluble, and the picrates of atropine, codeine, and thebaine readily so.

Thebaine from Codeine and Strychnine.—The picrate of thebaine is insoluble in water containing acetic acid, while the other two are soluble.

Neither potassium ferrocyanide nor ferricyanide gives any precipitates with atropine, brucine, codeine, veratrine, emetine, and sparteine. Thebaine is precipitated by potassium ferricyanide, but not quantitatively. The compound is readily soluble in HCl.

Sodium nitroprusside at first gives no precipitate with brucine and strychnine; gradually one is deposited, but the compound is soluble in pure water and fairly soluble in acidified water.

With veratrine there is an immediate precipitate, but this is not quantitative.

3. *Alkaloids, etc., not removed by Chloroform from Acid or Alkaline Hydroxide Solution.*—Morphine and narceine are removed by $CHCl_3$ containing alcohol, and strophantoin by a mixture of $CHCl_3$ and ether.

Narceine and strophantoin are insoluble in acidified tannic acid solution, while morphine remains in solution.

Further Methods of Separating Morphine and Narceine.—The compounds of morphine with picric acid and iodine are soluble in ammonium chloride solution, while the analogous compounds of narceine only dissolve in traces. Quantitative results are obtained by titrating the narceine by the iodine method described above under "Estimation of Alkaloids." The ammonium chloride exercises no influence on the iodine solution.

The picrates of both morphine and narceine are easily soluble in HCl, acetic acid, and ammonia.

For dissolving morphine picrate about 0.4 gramme of NH_4Cl is required for 0.1 gramme of the alkaloid salt. This solubility is due to dissociation of the compound.

With potassium chromate neutral solutions of narceine give no precipitate at first; but after standing some time one falls, and in from twenty-four to thirty-six hours the separation is quantitative. With morphine under similar conditions only traces of an insoluble compound appear.

Potassium bichromate at first gives no precipitate in neutral narceine solutions. On long standing there is a fine crystalline deposit, which is not quantitative. Morphine under similar conditions exercises a reducing action. There is no deposit in the presence of acid.

Strophantin is almost completely indifferent to most of the usual alkaloid reagents, and its separation is therefore easily effected.

In order to test the applicability of these methods to the separation and estimation of alkaloids in forensic cases; the author mixed weighed quantities of the under-mentioned alkaloids with various portions of the dead body, allowed these mixtures to remain for a time until putrefaction had set in, and recovered the alkaloids with the following results:

I. Taken, 0.1	gramme narceine	and 0.1	gramme codeine.
Found, 0.988	"	"	0.0982
II. Taken, 0.1	" strychnine	"	0.15 veratrine.
Found, 0.1006	"	"	0.1454
III. Taken, 0.1	" morphine	"	0.1 narceine.
Found, 0.0932	"	"	0.0991

C. A. M.

On the Determination of Formic Acid by Titration with Potassium Permanganate. H. C. Jones. (*Amer. Chem. Jour.*, 1895, xvii., 539-541.)—Péau de Saint-Gilles* was the first to determine formic acid by titration with potassium permanganate in the presence of an alkaline carbonate. Lieben† has confirmed this, using a more elaborate process. The method proposed by the author, and which was worked out in 1891, is on the same principle, but the procedure differs from that of Lieben.

The solution containing the formic acid is made alkaline with Na_2CO_3 , warmed, and an excess of standard permanganate added. All the formic acid is thus oxidized, and a precipitate of manganese hydroxide thrown down. The solution is acidified

* *Ann. Chim. Phys.*, 1859, lv., 374.

† *Monat. Chem.*, 1893, xiv., 746.

with H_2SO_4 , and a measured volume of oxalic acid run in until all the precipitate has dissolved and the permanganate disappeared. The excess of oxalic acid is then titrated with standard permanganate. A volume of oxalic acid equal to that taken is also titrated with the permanganate solution, and the difference between the result and the total permanganate used gives the quantity of permanganate required to oxidize the formic acid. The experimental results quoted agree well among themselves and with those obtained by other methods.

The author further shows that Saint-Gilles' statement that oxalic acid can be titrated in acid solution in the presence of formic acid is unreliable, since formic acid is also oxidized to some extent by the permanganate under these conditions.

C. A. M.

Estimation of Formic Acid. F. Freyer. (*Chem. Zeit.*, 1895, xix., 1184, 1185.)—Scala proposed to heat the neutral solution containing the formic acid with mercuric chloride, and to weigh the precipitated mercurous chloride. The author having occasion to determine the formate in a mixture of calcium acetate and formate, has devised the following method: The mixed calcium salts are distilled with dilute sulphuric acid in a current of steam until the distillate is no longer acid; an aliquot portion of the distillate is titrated with alkali to determine the total acid, whilst another portion is evaporated, if necessary, with excess of caustic soda to concentrate it, and is treated as follows: 10 to 20 c.c., containing about 0.5 gramme of formic acid, are heated for half an hour to an hour with 50 c.c. of a 6 per cent. solution of potassium bichromate and 10 c.c. of concentrated sulphuric acid in a flask provided with an inverted condenser. The liquid is now made up to 200 c.c., and the unaltered chromic acid determined in 10 c.c. of it. For this purpose, 1 to 2 grammes of pure potassium iodide, 10 c.c. of a 25 per cent. solution of phosphoric acid, and some water are added; and after five minutes the solution is diluted to about 100 c.c. with boiled water, and titrated with $\frac{N}{10}$ thiosulphate solution in the usual manner. The phosphoric acid is added according to Meineke's recommendation, and is for the purpose of rendering the change from the blue colour of the iodide of starch to the green of the chromium salt more visible; the commercial glacial acid may be dissolved in water, oxidized by potassium permanganate until it has a faint rose colour, and filtered.

The bichromate solution used for the oxidation is titrated in the same way. One mol. potassium bichromate is equivalent to three mols. formic acid.

The results quoted by the author show that the method is fairly accurate, both in the absence and in presence of acetic acid.

A. G. B.

Interpretation of some Results in the Analysis of Extracts of Fustic. C. S. Boyer. (*Jour. Amer. Chem. Soc.*, 1895, xvii., 518-520.)—The method of analysis employed was as follows: Water was estimated by drying 5 to 6 grammes of the extract in the water-oven until the weight was constant. Two to four grammes of this dry powder were completely exhausted with absolute alcohol in a Soxhlet

extractor, and the residue left on distilling the alcohol from the extract dried and weighed. The residue was treated with boiling water and tested for *morin* and *maclurin* (*morin-tannin*) in separate portions, the former by adding a few drops of aluminium sulphate, and the latter by adding ferric chloride. In the samples examined by the author, the result was negative in each case. The residue from the alcoholic extract was ignited in a platinum crucible, and the ash subtracted from the matter soluble in absolute alcohol. The ash percentage was determined on 5 grammes of the powder. The results obtained were:

	I. On dry basis.		II. On dry basis.		III. On dry basis.		IV. On dry basis.		V. On dry basis.	
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Water	67.09	—	50.42	—	36.48	—	49.62	—	6.18	—
Organic matter soluble in absolute alcohol	31.34	95.23	38.57	77.79	52.85	83.20	47.41	94.11	75.55	80.53
Ash	0.52	1.58	5.54	11.18	1.24	1.95	1.05	2.08	7.62	8.12
Organic matter insoluble in alcohol	1.05	3.19	5.47	11.03	9.43	14.85	1.92	3.81	10.65	11.36
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

The chief mineral constituents of fustic are lime and magnesia, and the amount of these forms one of the guides in determining the method of extraction. The more pressure used the higher, within certain limits, the ash percentage, and the larger the amount of lime and magnesia in the ash. The extracts contained the following quantities of lime and magnesia: I. 41.16 per cent., IV. 46.83 per cent., II. 68.12 per cent., V. 62.15 per cent. of the total ash. The presence of quercitron-bark extract as an adulterant may often be misleading. This was tested for by a series of dye-tests depending on the different affinities of the colouring matter of bark and fustic for alum and tin mordants, but the results were negative.

The conclusions arrived at were that I. was made entirely by the "open extraction" method; II. by the "closed extraction" method, using five to eight pounds pressure; and that III. was extracted in open vessels, but that the change of "waters" was done under pressure. The latter inference was drawn from the fact that the percentage of ash was low, while the "extractive matter" was high; and the only way this could happen would be by opening up the fibre of the wood, as in open boiling, and then applying pressure. The conclusions were subsequently confirmed by experiment.

The origin of the other two samples was known. IV. was made in an open extractor with boiling water. Eight "waters" were taken off, each having remained fifteen minutes in contact with the wood. Sample V. was extracted under five pounds pressure, using seven "waters," and the weak liquor being evaporated to dryness.

C. A. M.

A New Method of Analysing Fats and Hydrocarbons. M. Crismer. (*Bull. de l'Assoc. belge des Chimistes*, ix., 1895, pp. 71, 72.)—The method depends on a new physical constant which the author calls the *critical temperature of dissolution*. Several drops of the melted and filtered substance are introduced into a small tube

several millimetres in diameter by means of a capillary pipette. A slightly greater volume of alcohol of known density is then added, and the tube sealed and fixed by a platinum thread to the bulb of a thermometer. It is then heated in a bath of sulphuric acid until the meniscus separating the two layers becomes a horizontal plane. At this point the thermometer is withdrawn from the bath and turned sharply two or three times until the liquid becomes homogeneous, after which it is replaced and the temperature allowed to fall slowly, the thermometer and tube being constantly shaken. The temperature at which a marked turbidity is produced in the liquid is the critical temperature of dissolution.

The conclusions arrived at by the author are: (1) Substances of the same nature have practically the same critical temperature of dissolution. Thus, fourteen butters of guaranteed purity from different sources varied from 98° to 102° , the mean being 100° C.; samples of margarine gave a critical temperature of from 122° to 126° ; cocoanut-oil from 71° to 75° ; cocoa-butter, 126° ; pure beeswax from various sources, 129° to 133° ; ozokerite, 175° ; Carnauba-wax, 154° ; paraffins, according to their constitution and melting-points, 140° to 160° ; and essence of terebenthene, 14° .

(2) The critical temperature of a mixture is the arithmetical mean of those of its constituents.

C. A. M.

A New Method for the Quantitative Estimation of Starch. M. Dennstedt and F. Voigtländer. (*Forsch. Ber. üb. Lebensm.-Hamburg Chem. Staats Lab.*; through *Chem. Cent.*, 1895, pp. 322, 323.)—According to the authors the blue colour produced by iodine in starch solutions is in direct proportion to the amount of starch, and on this they have based a calorimetric method of estimation. On boiling a little starch in a large quantity of water the starch granules are in so fine a state of division that it behaves like a solution, while the starch cellulose falls to the bottom. For wheat-starch the relation between them appears to be constant (90.5 : 100). In order to obtain a solution containing a known quantity a pure starch is selected, and the moisture, ash, protein and fat determined and deducted from the amount taken.

A quantity corresponding to 0.5 gramme of starch, as determined by these calculations, is weighed out to the fourth place of decimals, and boiled with a litre of water in a 2-litre flask. On cooling the whole is made up to a litre, the starch cellulose allowed to settle, and 5 c.c. of the supernatant liquid poured into a series of similar cylinders containing 100 c.c. and graduated in $\frac{1}{2}$ c.c.s. One drop of a solution of I in KI is then added to each, and all are made up to 100 c.c. The substance to be examined is treated in the same way, 0.5 gramme being weighed out after the determination of the moisture, etc., and the solution made as in the case of the pure starch. The colour produced by the known solution of starch is then matched by the unknown, and the mean of several determinations taken. As it is more easy to judge a colour between a lighter and a darker, it is advisable to have cylinders, some containing 4.9 c.c. of the known solution and others 5.1 c.c., and to place the one to be examined between them. A constant temperature should be observed, and the solutions always freshly prepared. A violet colour, instead of a blue, may be given with fine meal. This is remedied by stirring the weighed

amount with alcohol, and, after it has stood some time, filtering with the aid of a filter-pump through a starch-free filter. The filter is washed with alcohol, ether, and alcohol again, and is introduced together with the meal into the flask.

C. A. M.

Detection of Naphthol. H. Spinette. (*Bull. de l'Assoc. Belge des Chim.*, ix., 1895, pp. 50-52.)—The use of naphthol as an antiseptic in food products is not of frequent occurrence. The author has only once met with it. This is probably owing to its small solubility in water, and to the taste it communicates. In spite of these drawbacks, however, it may be met with in beverages, and especially in substances sold as preservative agents. It is, therefore, advantageous to determine the best method of isolating and identifying it. Two varieties are known— α -naphthol and β -naphthol. The former crystallizes in colourless needles, fuses at 94° , and boils at 280° C. It has a strong odour of phenol. β -naphthol, or iso-naphthol, forms rhombic colourless crystals, fuses at 122° , and boils at 290° . It is almost without smell, and has a bitter, burning taste. It is but little soluble in water, but dissolves readily in alcohol, ether, chloroform, and solutions of the alkalies.

The detection of the phenols in general has been studied by Desquelle.* In his method the substance to be examined is shaken gently with half its volume of chloroform, the chloroform drawn off by means of a separating funnel, and a small piece of caustic potash added to it. Characteristic colours are obtained, varying with the nature of the phenol.

Phenol	Rose.	Beta-naphthol	Greenish-blue.
Resorcin	Rose.	Pyrogallol	Violet.
Hydroquinone	Golden yellow.	Cresol	Violet.
Alpha-naphthol	Sky-blue.	Guaiacol	Violet-rose.

Yvon identifies naphthol by the rose coloration given by it with either of the following reagents:

1. Mercuric nitrate 5 grammes.
Nitric acid 15 "
2. Saturated solution of potassium nitrate 10 "
Sulphuric acid 5 "

The author found that Desquelle's method did not always give conclusive results, and so modified it by making the tests on the residue left on the spontaneous evaporation of the chloroform.

The following test may also be applied: The residue is dissolved in 5 c.c. of caustic soda solution, and an aqueous solution of 0.05 gramme of aniline sulphonic acid added. This is made by dissolving the acid in 5 c.c. of normal soda solution, and then adding 5 c.c. of normal H_2SO_4 and 0.02 gramme of sodium nitrite.

With alpha-naphthol a blood-red colour is obtained; with beta-naphthol a yellowish red.

C. A. M.

A New Method of estimating Indigotin. Josef Schneider. (*Zeit. Anal. Chem.*, 1895; *Drittes Heft.*, pp. 347-354.)—All the methods in use for estimating indigotin are more or less unsatisfactory, especially when used to determine it in

* *Repertoire de Pharmacie* 1890, p. 101.

coloured fabrics. The errors in most of them were pointed out by Von Cochenhausen,* while Ulzer† in turn showed that the process of Von Cochenhausen and Hönig was very liable to error, since on continued boiling with aniline a considerable amount of indigo was lost, for which no correction was possible. The author considered the method of W. Stein‡ as being the most promising. In this 0.2 gramme of indigo is boiled for fifteen minutes with 20 c.c. of an animal oil with a boiling-point above 180° C. The liquid is poured on to a filter, and the boiling repeated with 10 c.c. of oil (at least three times), until the indigotin is completely extracted. The cooled filtrate is then well shaken with twice its volume of ether, allowed to stand for an hour, filtered through a weighed filter, the deposit washed with ether, dried at 100°, and weighed.

According to the author, there are three main objections to this process.

1. Indigo only dissolves in considerable quantities at the boiling temperature of the oils, and only in those which boil above 180°. As the temperature falls, the solubility declines very rapidly. It is, therefore, necessary to filter at a high temperature.

2. Indigotin cannot be completely precipitated from its solution in animal oils.

3. As in the case of aniline extraction, there is a loss of indigo on heating with animal oils, this being greater the more oil and indigo are used, and the longer the boiling is continued.

The principal novelties in the author's process are the use of a special extraction apparatus and of a solvent (naphthalene) which does not act on indigotin.

The naphthalene (50 grammes) is boiled in an Erlenmeyer flask, through the cork of which passes a tube 15 millimetres in width and a metre long. In the side of the tube within the flask there is an opening, and the bottom of the tube is contracted and slightly bent. The indigo ($\frac{1}{2}$ to 1 gramme) is mixed with glass wool, placed in a paper coil surrounded by a linen one, and suspended below the bottom of the tube in the flask, so that the naphthalene falls into the coil. In order that accurate results may be obtained, the naphthalene must be free from ash. The boiling must be continued until the drops falling from the coil are quite colourless. On cooling, the naphthalene solution of indigotin is decomposed with ether, filtered, and weighed, as in Stein's method. The correction to be applied for the indigo decomposed and remaining in solution depends on the relative quantities of naphthalene and indigo used, on the duration of the extraction (with 1 gramme of indigo usually 5½ hours), on the manner of heating, and on the possibility of overheating. In order to determine it, the indigotin obtained on the filter should again be extracted with naphthalene under exactly similar conditions, and the loss on again weighing the indigotin gives the necessary correction. With 50 grammes of commercial white naphthalene which was not quite pure, the loss of indigotin on heating over wire gauze was 1 to 4 milligrammes, corresponding to a correction of +0.1 to 0.4 per cent., with 1 gramme of indigo. By using purified naphthalene and heating on an oil bath, the loss would certainly have been less.

* *Leipziger Monatshefte für Textilindustrie*, 1888.

† *Mitth. des technol. Gewerb.*, Vienna, 1891.

‡ *Die Prüfung der Zeugfarben und Farbmaterialien*, 1874.

Care must be taken to have the apparatus and the indigo completely dry, in order to avoid the risk of explosions. C. A. M.

Amalgamated Aluminium as a Neutral Reducing Agent in Presence of Water. H. Wislicenus and L. Kaufmann. (*Ber.*, 1895, xxviii., 1323.)—An amalgam of aluminium containing as little as one atom of mercury to forty-five of aluminium decomposes water with greater energy than the well-known amalgam of sodium, while the mercury serves only as a "catalytic" agent. On alcohol and ether it has no action, and may be used, therefore, to remove every trace of moisture from these bodies. Although the evolution of hydrogen is somewhat stormy, the action can be perfectly regulated by cooling the liquid. As a reducing agent, the substance to be treated is dissolved in ether or absolute alcohol (aqueous alcohol or even water may be used if necessary), an excess of the amalgam added, and water dropped in with constant agitation. The action is usually complete in a very short time, and the alumina may be filtered off or removed by the pump, and will rarely be found to retain any of the organic substance. Any other indifferent solvent or mixture of solvents may be employed so long as they dissolve traces of water.

The amalgam is prepared by treating aluminium turnings (freed from oil) with caustic soda until they are attacked. They are rinsed in water, and a 0.5 per cent. solution of mercuric chloride allowed to act for one or two minutes. The operations are repeated, and the black amalgam is washed rapidly in water, alcohol, and ether, and preserved, if necessary, under petroleum spirit. This process is specially adapted to the reduction of nitro-compounds. Its use on a commercial scale is the subject of a patent. F. H. L.

The Action of Thiocetic Acid on Various Metallic Solutions in the Cold. N. Tarugi. (*Gazz. Chim. Ital.*, 1895, xxv. [I.], 341; through *Chem. Zeit. Rep.*, 1895, 201.)—When employing thiocetic acid to replace sulphuretted hydrogen in ordinary analysis, it is necessary to conduct the precipitation in hot solutions, in which case only are pure sulphides thrown down. By adding this reagent to cold neutral mercuric chloride solution, washing and drying the precipitate in the dark, extracting with carbon disulphide, and finally drying over sulphuric acid *in vacuo*, a white thio-chloride ($2\text{HgS}, \text{HgCl}_2$) is obtained, soluble only in aqua regia. A similar thio-nitrate may be prepared, which is blackened by caustic soda or ammonia. The white precipitate obtained from an aqueous mercuric acetate solution could not be analysed owing to its instability, but by employing an alcoholic solution of the same salt a white crystalline powder turning to yellow was obtained, consisting of a mixture of normal and basic mercuric thiocetate separable by boiling chloroform, in which only the neutral salt is soluble. On cooling, this is thrown down as white, mother-of-pearl-like crystals, which, when pure, are unchanged by light and air, and are soluble in warm petroleum spirit, less so in alcohol and carbon disulphide. It is unchanged by boiling water or by dilute cold hydrochloric acid, but warm dilute hydrochloric or cold dilute sulphuric acid converts it into the previously described thio-chloride. The basic mercuric thiocetate is an amorphous orange powder in-

soluble in hydrochloric or nitric acid and ammonium sulphide, but dissolved by aqua regia.

Cold neutral copper sulphate yields a green precipitate, probably a mixture of sulphide and thiacetate. Cadmium solutions give a white amorphous precipitate of thiacetate, which, when moist, is altered by exposure to light, is soluble in warm dilute acids, and by the addition of ammonia or ammonium sulphide or boiling with water is changed into the sulphide. Lead solutions also yield a mixture of thiacetate and sulphide, the former being soluble in boiling water, and forming white needles permanent in light and insoluble in alcohol, chloroform, and petroleum spirit. Nitric acid converts it into lead sulphate; ammonia, caustic soda, and ammonium sulphide into lead sulphide. Silver salts give a reddish precipitate with this reagent, but it is too unstable to allow of analysis.

For the preparation of thiactic acid for use in chemico-legal investigations, R. Schiff (*Ber.*, 1895, xxviii., 1204) recommends 300 grammes of phosphorus pentasulphide, 150 grammes of broken glass, and 300 grammes of glacial acetic acid to be gently warmed together in a 2-litre flask fitted with a thermometer and an inverted condenser. When the vapours reach 103° the operation is stopped and the product rectified several times, the portion distilling between 92°-97° being kept. Prepared as above, the reagent is absolutely free from arsenic. F. H. L.

A New Method for the Separation of Copper and Cadmium in Qualitative Analysis. A. S. Cushman. (*Amer. Chem. Jour.*, 1895, xvii., 379-383.)—It is well known that cadmium combines with the chlorides of the metals of the alkalis and alkaline earths, forming, according to Hauer, compounds of the general formulæ $4\text{RCl}, \text{CdCl}_2$; $2\text{RCl}, \text{CdCl}_2$; $\text{RCl}, \text{CdCl}_2$. With ammonium chloride the compound $2\text{NH}_4\text{Cl}, 2\text{CdCl}_2, \text{H}_2\text{O}$ has been obtained. These double chlorides are formed when cadmium sulphide is treated with dilute hydrochloric acid in the presence of alkaline chlorides, the reaction being $\text{CdS} + 2\text{RCl} + 2\text{HCl} = \text{CdCl}_2, 2\text{RCl} + \text{H}_2\text{S}$. This property of cadmium sulphide is used by the author as the principle of a delicate test for cadmium. If to 2 c.c. of a solution containing a small amount of cadmium, 10 c.c. of a saturated solution of NaCl and a few drops of dilute hydrochloric acid be added, hydrogen sulphide produces no precipitate even when passed in to saturation. If a few drops of dilute ammonia be allowed to run down the side of the tube, a yellow ring forms at the junction of the liquids. This test was sensitive with a solution containing less than 0.01 milligramme of cadmium, a perceptible ring appearing after standing half an hour.

In separating the sulphides of copper and cadmium, the latter entirely dissolves on adding a strong solution of salt and a little hydrochloric acid. On diluting the filtrate and adding hydrogen sulphide, the cadmium is re-precipitated. Care must be taken that lead and bismuth are absent, since the sulphides of these metals are also soluble in strong acidified solutions of the alkaline chlorides. C. A. M.

The Estimation of Citrate-soluble Phosphoric Acid in Thomas' Slag.
P. Wagner. (*Chem. Zeit.*, 1895, xix., 1419.)—When this process was first devised it was intended only as a rough test for different makes of slag, and to serve as a means of detecting adulteration. The material was then of very irregular composition, and samples, all containing about the same amount of total phosphoric acid, differed very widely in their solubility in ammonium citrate solution, and consequent manurial value. The position has now changed. The manufacture of the slag has been greatly improved, and the process of examination has proved itself so exact, that it has been adopted in Germany as the standard method for the commercial valuation of basic slag. It is important, however, in carrying out the process to adhere strictly to all the details mentioned, as any departure from the regular routine in strength or temperature of solutions, or time of digestion, etc., may be followed by grave errors.

Strong Ammonium Citrate.—1,500 grammes of pure crystallized citric acid are dissolved in about 2 litres of water, $3\frac{1}{2}$ litres of 8 per cent. ammonia added, and, when cold, the liquid is made up to 8 litres. In a small quantity (2.5 c.c.) of this solution the ammonia is determined by distillation with magnesia, and, finally, enough ammonia and water are added to the bulk to make 10 litres of solution, containing exactly 279.3 grammes of ammonia. A *dilute citrate solution* is prepared by mixing two volumes of this solution with three volumes of water.

Molybdate Solution.—Prepared either by dissolving 150 grammes of ammonium molybdate in water, or 125 grammes of molybdic acid in 100 c.c. of water and 300 c.c. of 8 per cent. ammonia. Four hundred grammes of ammonium nitrate are added, the solution made up to one litre, and poured into an equal volume of nitric acid (specific gravity 1.19). After standing twenty-four hours at 35°C., the whole is filtered.

Magnesia Mixture.—110 grammes of pure magnesium chloride and 140 grammes of ammonium chloride are dissolved in 700 c.c. of 8 per cent. ammonia and 1,300 c.c. of water, the solution being filtered after standing several days.

Five grammes of the slag just as received are put into a half-litre bottle and filled up to the mark with the dilute citrate solution at a temperature of $17\frac{1}{2}$ ° C.; a rubber cork is inserted in the neck, and the bottle shaken mechanically for half an hour (if a revolving apparatus be employed, the speed should be thirty to forty revolutions per minute). The liquid is then immediately filtered as rapidly as possible, the filtrate being returned to the filter if not clear. Fifty c.c. are measured into a beaker, 100 c.c. of the molybdate solution added, and placed for ten to fifteen minutes in a water-bath at 80° to 95°. When cold, the precipitate is filtered off, washed with 1 per cent. nitric acid, and dissolved in 100 c.c. of cold 2 per cent. ammonia (should this solution not be perfectly clear, the analysis is useless). Fifteen c.c. of the magnesia mixture are next added drop by drop, with constant stirring, the vessel covered over and put aside for two hours; the precipitate is then filtered, washed with 2 per cent. ammonia, and ignited as usual. The temperature of agitation ($17\frac{1}{2}$ ° C.) is specially important, and must be maintained as accurately as possible during the whole of the thirty minutes.

F. H. L.

CORRESPONDENCE.

To the Editors of THE ANALYST.

SIRS,—In reply to Dr. E. Frankland's letter in this month's *ANALYST*, permit me to say that I had not seen the Registrar-General's annual summary referred to, and, therefore, was not aware that Dr. Frankland had suggested a specific cause for the discrepancies in question. The suggestion does not appear to be quite satisfactory, because the companies' chemist's organic C is very frequently about twice as much as Dr. Frankland's, whereas, if the suggested cause of the discrepancies in the N is the true one, it should be less. It is, however, remarkable that such an error could have been allowed to exist for so long a time without rectification.

I may mention that I brought the matter of these differences in analytical results before the Royal Commission on Metropolitan Water Supply, 1892, and handed in a statement, a copy of which I enclose. I also discussed the subject in a paper contributed to the Sanitary Congress at Portsmouth in 1892, a copy of which is also enclosed. Dr. E. Frankland's statement refers only to the results obtained by the chemists acting for seven of the London Water Companies. The chemist for the other company—the Kent—was the late Dr. A. Bernays, and I know from personal experience that he strictly adhered to all the directions given by Dr. Frankland in his book.

In my paper I give the results obtained by the late Dr. Bernays during 1891, and the differences between these and Dr. E. Frankland's are enormous, and far exceed those of any of the others.

Since February, this year, the results of an analysis of the Kent company's water by Dr. P. Frankland have been published in the monthly reports of the official water examiner. The organic C and N figures in these analyses differ vastly from those obtained by Dr. E. Frankland from the same company's water, as will be seen below :

					Dr. P. Frankland.		Dr. E. Frankland.
<i>February...</i>	...	Organic C	·0557	...	·04
"	...	" N	·0157	...	·005
<i>March</i>	...	" C	·0557	...	·038
"	...	" N	·0157	...	·005
<i>April</i>	...	" C	·0485	...	·023
"	...	" N	·01	...	·005
<i>May</i>	...	" C	·0485	...	·024
"	...	" N	·0157	...	·008
<i>June</i>	...	" C	·0471	...	·024
"	...	" N	·0114	...	·006

It is curious that the N in Dr. P. Frankland's results for February and March should be three times, and in April, May, and June twice as much as Dr. E. Frankland obtained.

There are many other discrepancies to be found in the published results of the analyses by the companies' chemists as compared with Dr. E. Frankland's, since they have added the "inch of copper oxide in front" to the contents of the combustion-tube ; but I think the above are sufficient to show that the process does not in practice give reliable results even in the hands of the most experienced operators.—I am, sir, your obedient servant,

W. C. YOUNG.

LONDON, August 23, 1895.

THE ANALYST.

OCTOBER, 1895.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

DUCLAUX'S METHOD FOR THE ESTIMATION OF "VOLATILE FATTY ACIDS," THE LAWS GOVERNING "VOLATILITY" DEDUCED THEREFROM, AND THEIR APPLICATION TO ANALYSIS, MORE ESPECIALLY TO THAT OF BUTTER.

By H. DROOP RICHMOND.

(Concluded from p. 198.)

PART II.—RESULTS OBTAINED IN THE PRESENCE OF A SUBSTANCE IN WHICH VOLATILE FATTY ACIDS ARE SOLUBLE AND IMMISCIBLE WITH WATER.

IN a method which depends largely on the laws of volatility—the Reichert process—we have conditions which are different to those under which the foregoing experiments were made. In this a mixture of butyric, caproic, and other acids are distilled from an aqueous solution, with which is mixed a quantity of insoluble fatty acids, which Wollny (ANALYST, xiii., 11) has shown dissolve some of the volatile acids. I have already shown (*loc. cit.*) that butyric acid is not held back by insoluble fatty acids, as Wollny supposes.

I have made several practical distillations of butter, with very concordant results, and find that the relation between the volatile acids in the distillate and the volume of the fraction cannot be expressed by a formula of the type $100 - y = \frac{(100 - x)^a}{100^{a-1}} \times k^{-z}$.

The mode of experiment was as follows: 2.5 grammes of butter were saponified by 1 c.c. 50 per cent. soda solution, and 5 c.c. of glycerol or alcohol; if alcohol were used, it was distilled off under reduced pressure, and 55 c.c. water added, but if glycerol, 50 c.c. of water were taken; 20 c.c. dilute sulphuric acid (25 : 1000) was measured into the flask, the fatty acids melted under a reflux condenser, and when this was attained, distillation was proceeded with. Fractions 0–20 c.c. (or 0–22.25) 20–40 c.c., 40–50 c.c., and 50–60 c.c. were collected, then successive portions of 10 c.c. of well-boiled water were added, and distilled till 10 c.c. only took one drop of $\frac{N}{10}$ alkali solution for neutralization; this was taken as the fraction 60–75 c.c., and the sum of the volatile acid in the fractions as the total in the butter.

Up to a certain point they agree well with my butyric acid curve, but at the end of the distillation the want of agreement is very marked.

The following results have been obtained :

Vol. of Distillate. Total = 75 c.c.	Percentage of Acid Distilled.	Acid Distilled × 1.072.	Calc. for Butyric Acid.
20	46.3	49.6	49.4
22.5	50.8	54.4	53.4
40	74.9	80.3	80.7
50	84.4	90.5	90.5
60	91.1	97.6	96.7
75	100.0	107.2	100.0

In the third column the figures are multiplied by 1.072, to show more clearly the agreement with the numbers calculated for butyric acid. The volatile acids of butter appear to distil as a mixture of butyric acid with some other acid volatile towards the end. I have already suggested the presence of lactic acid, but I have been unable to prove its presence. That the apparent divergence from the laws of volatility is due to the presence of the insoluble fatty acids is shown by the numbers obtained on redistilling the distillate from a butter, which Duclaux has shown to distil as a mixture of butyric and caproic acids.

I have shown in the Reichert-Wollny process, when pure butyric acid is used in place of butter, that 96.9 per cent. are distilled when 110 c.c. out of 140 c.c. have passed over ; when 4.4 grammes of well-washed fatty acids were added, 97.2 per cent. were distilled, so that about 97 per cent. of the total butyric acid present should be distilled in the Reichert-Wollny process.

I find four experiments by Wollny (*loc. cit.*), which I have corrected slightly by deducting .22 c.c. for each 110 c.c. of water added and distilled (a figure deduced from my own experiments), and quote four of my own :

		R.-W. Figure.	Total.	Per Cent. Distilled.
Wollny	...	27.94	31.33	89.2
"	...	26.52	30.48	87.0
"	...	27.77	31.51	88.1
"	...	31.07	35.80	86.8
Richmond	...	27.7	32.1	86.3
"	...	29.0	33.4	86.8
"	...	29.1	33.6	86.6
"	...	29.0	33.2	87.4

Thus we see that in the Reichert-Wollny process only about 87 per cent. of the total acid is found in the distillate.

Now, Duclaux has shown that when γ_{11} are distilled, a proportion approximating to the Reichert-Wollny quantities, the composition of the volatile acids in the distillate is approximately 1.8 mols. butyric acid to 1 mol. caproic acid.

I have already shown that 97 per cent. of the butyric acid should distil, so that there should exist in the butter $\frac{1.8}{.97} = 1.856$ mol. butyric acid for 1 mol. caproic acid in the distillate.

To each molecule of caproic acid in the distillate there are 2.8 molecules of volatile acids ; as only 87 per cent. of the acid is found in the distillate, there should be $\frac{2.8}{.87} = 3.218$ molecules of volatile acids in the butter ; of this, it has just been

shown that 1·856 molecules are butyric acid, and therefore 1·362 molecules of caproic acid exists in the butter for each molecule in the distillate. The volatile acids of butter contain 57·7 molecules of butyric acid to 42·3 molecules caproic acid; and from the table given above the following apparent rate of distillation of caproic acid can be deduced :

Vol. of the Distillate. (Total 75 c.c.)	Per Cent. Caproic Acid Distilled.	Calc. for Caproic.	Composition of Vapour.	Composition of Liquid.
20	42·1	73·1	139·5	34·9
22·25	47·3	78·4	124·0	31·0
40	66·9	97·3	82·3	20·6
50	76·1	99·0	62·0	15·5
60	83·5	99·9	65·0	16·4
75	100·0	100·0	—	—

The composition of the vapour is obtained by interpolation, and the composition of the liquid deduced from this by dividing by 4, which, as was shown in Part I., is the ratio between the composition of the liquid and the vapour. From the composition of the liquid and the quantity left in the retort, the quantity of acid dissolved in the liquid can be calculated, and the remainder will be the quantity dissolved by the fatty acids.

Vol. of the Distillate.	Per Cent. Acid in Liquid.	Per Cent. Acid in Fatty Acids.	Composition of Liquid.	Ratio { Per Cent. in Fatty Acids. Composition of Liquid.
20	25·6	32·3	34·9	·9
22·25	21·9	30·8	31·0	1·0
40	10·5	21·6	20·6	1·0
50	5·2	18·7	15·5	1·2
60	3·3	13·2	16·4	·8
75	—	—	—	—

The figures in the fifth column, which are, of course, relative and not absolute, are approximately constant, and when it is considered that they are subject to the following errors—experimental error, error in computation of ratio of butyric acid to caproic acid, errors due to condensation, error due to interference of other acids, *e.g.*, caprylic, and errors in interpolation—the agreement is remarkable.

An extension of Henry's law to express the distribution of a gas between a space and two immiscible liquids shows us that the quantity in the space is an inverse function of the vapour pressure and the solubility in each liquid, and of the bulk of the liquids.

The laws of volatility in the presence of a third body become very complex, and it is practically impossible to obtain any useful information from fractional distillations of butter, unless the whole of the volatile acid be first distilled, and its properties studied in the absence of a second liquid.

To show the nature of the errors thus obtained, Duclaux calculates from his figures that the ratio of the total butyric acid to the total caproic acid in butter is 2 mols. to 1 mol., while from the same figures I calculate the proportion to be 1·36 mols. to 1 mol., and Viollette finds the mean proportion to be 1·65 mols. to 1 mol.

The Viscosimetrical Examination of Butter for Foreign Fats. Dr. Newman Wender. (*Journ. Amer. Chem. Soc.*, xvii., 1895, pp. 719-723.)—It has been well established that there is a definite relation between the chemical composition and the viscosity of liquids, and hence the determination of the rate of transfusion has been found of practical value in the examination of many substances—notably, oils and beer. To the usual apparatus employed for this purpose the author has added the “fluidometer.” (Manufactured by Max Kaehler and Martini, Berlin.) This consists of a U-shaped capillary tube, with the limbs enlarged and divided in such a way that the one holds 10 c.c. and the other 2 c.c. of the liquid. The viscosity is calculated from the time taken by the liquid to flow from the wider limb into the smaller one, which is placed somewhat lower. The apparatus, which is inexpensive, is easily and quickly cleaned.

The researches of Graham (*Liebig Annal.*, 123, 90), confirmed by those of Pribram and Handl and by those of Gartenmeister (*Zeit. Phys. Chem.*, 6, 524), have shown that the viscosity of a liquid increases with the molecular weight. This is evident from the following table :

Fatty Acids.	Molecular Weight.	Specific Gravity at 20° C.	Boiling-Point.	Pribram and Handl.			Gartenmeister.
				Specific Viscosity at 10° C.	Specific Viscosity at 30° C.	Specific Viscosity at 50° C.	Absolute Constant of Friction at
Propionic ...	74	0.9929	140.7	70.3	51.5	49.9	— 0.1128
Butyric ...	88	0.9580	163.0	110.2	77.4	57.6	— 0.1634
Valerianic ...	102	0.9386	184.0	152.4	103.3	71.5	— 0.2279
Capronic ...	116	0.9279	199.7	222.2	139.7	97.8	— 0.3263
Heptylic ...	130	0.9163	223.0	—	—	—	— 0.4440
Octylic ...	144	0.9115	237.0	—	—	—	— 0.5860
Nonylic ...	158	0.9053	253.0	—	—	—	— 0.8480

Hence margarine with glycerides of a high molecular weight will also have a higher viscosity than butter which contains volatile glycerides in addition. The determinations of Gaselli and Carcano (*Centrbl. Agr. Chem.*, 1894, 838), showed that the molecular weight of pure butter lay between 695 and 715, while that of margarine varied from 780 to 883.

Killing (*Zeit. angew. Chem.*, 1894, 693; *ANALYST*, xx., 66 and 94) confirmed the relationship of the molecular weight to the viscosity in the case of butter and margarine, though the different samples of the latter showed very variable values.

Since, according to Traube (*Berliner Ber.*, 1886, 871), the relation between molecular weight and viscosity is not affected by solvents, the author uses for his “fluidometer” a solution of the melted fat in chloroform, and thus avoids the high temperatures necessary to maintain the fat at the melting-point. The viscosity of the solvent is also to be taken into account. By this method samples of butter from different sources required in the mean 20.04 seconds at 20° C. for efflux. The time required by the solvent was set at 100, and the calculation based on this.

AVERAGE RESULTS.

Viscosity value for pure butter, 344.30. Time, 68.8.

„ „ margarine, 373.20. „ 77.4.

Every degree of temperature above 20° C. decreases the efflux time by 1.45 seconds, but the time the solution of the fat is allowed to stand does not influence the result. A decreasing temperature retards the efflux by an average of 1.43 seconds for every degree.

In the author's opinion, the viscosimetrical examination of butter is as conclusive as any other physical test, but when margarine is present in only a small quantity its detection cannot be effected in this way. C. A. M.

Test for Distinguishing between Butter and Margarine. (*Pharm. Ztg. Berlin*, through *Seifenfabrikant*, vol. xv., p. 579.)—J. Rolfs, on washing out the sodium butyrate obtained by treating rancid butter with sodium bicarbonate, found that the butter became pale and crumbly in appearance. The same test applied to margarine produced no change, and the presence of this adulterant in butter prevented the alteration signalized in the case of butter alone.

On replacing the sodium compound by potassium carbonate, the following results were obtained, working with 2 to 5 grammes of butter, and employing 20 grammes of water in the washing :

1. The persistent emulsion produced by pure butter is completely dissolved to a clear solution by an equal volume of ether, the line of demarcation between the ethereal and aqueous liquids being sharply defined.

2. With butter and margarine in equal parts an emulsion is obtained, but the lower part of the ethereal solution contains flocculent matter in suspension, and only clarifies by subsidence after prolonged rest.

3. Where the proportion of margarine is raised to three times that of butter, the production of an emulsion is tedious, and requires long-continued agitation. In this case the emulsion is broken up by the addition of half the usual amount of washing water, and the ethereal layer is almost completely filled with suspended matter.

4. On the other hand, neither emulsion nor ethereal solution can be obtained from margarine alone.

The circumstance, therefore, that pure butter, when treated with an alkaline carbonate, will form, with water, an emulsion soluble in ether, whereas margarine gives no such results, its presence in a mixture of the two bodies being revealed by the precipitation of flocculent particles, affords a rapid and reliable means of distinguishing between them, and of detecting adulteration by the last named. C. S.

An Abnormal Butter. J. Samelson. (*Chem. Zeit.*, 1895, xix., 1626.)—A sample of Bavarian butter was recently examined by the author, and gave the following results : Reichert-Meissl number, 21.6 ; Hehner's number, 89.2 ; saponification number, 216.0 ; and iodine number, 42.5. It was therefore returned as adulterated with foreign fat. A thorough investigation followed, and the butter was proved conclusively to be perfectly genuine. Further samples made from the milk of the same cow, and analysed by Soxhlet, gave similar figures. F. H. L.

Baudouin's Reaction for the Detection of Sesame in Olive-Oil. E. Carlinfanti. (*Selmi*, 1895, v., 49; through *Chem. Zeit. Rep.*, 1895, 215.)—The author finds that the red colour which appears even when the olive-oil examined is pure may be removed by shaking the acid with three times its volume of water; while if only 0.5 per cent. of sesame-oil be present, the colour is not affected by this treatment.

F. H. L.

Lard. M. Mansfield. (*Ztschr. Nahrungsm. Unters. Hygiene*, 1895, ix., 200; through *Chem. Zeit. Rep.*, 1895, 215.)—The author does not believe that commercial lard varies so much in its iodine number as is often reported. He obtains regularly a value of 59 to 62. To test for beef tallow, benzene is the best solvent, pure lard crystallizing therefrom in needles either singly or in bundles, while in the presence of the beef tallow cauliflower-like masses of crystals are obtained. For the detection of further adulteration, the iodine number, crystallizing and solidifying points of the fatty acids may be determined; but for an approximately quantitative idea of the composition of the fat it is necessary, by preparation of the zinc salts and extraction with ether (in which they alone are soluble), to separate the liquid acids. These, as obtained from lard, have an iodine number of 92 and a rotation value of 44 to 45. Vegetable oils subjected to this treatment show a much higher iodine number.

F. H. L.

The Examination of Lard. A. Goske. (*Chem. Zeit.*, 1895, xix., 1043.)—For the detection of tallow in lard, the author still holds that microscopic examination is the surest plan, but the appearance of the crystals obtained from the steam lard and neutral lard of America, is somewhat different from those of ordinary butchers' lards. Not more than 1 gramme of the material is dissolved in 10 c.c. of ether and allowed to crystallize at 12° to 13°C., when pure steam lard will be found to assume the form of sharply defined plates mixed with a few bundles of needle-shaped crystals. If beef-tallow be present, no plates will be visible, the crystals being needles radiating from centres. (They consist of the beef stearin, the pork stearin remaining dissolved.) Butchers' lard also crystallizes in needles, but these are much larger, and more matted together. The addition of either oleomargarine or mutton tallow influences somewhat the appearance of the crystals, but the change can hardly be described.

In testing for the addition of oil, the author has partly given up the use of phospho-molybdic acid, as the indications vary according to the age of the material: he finds, however, that Becchi's silver test in the form recommended by the Italian Commission is quite reliable. Two solutions are prepared—(1) 1 gramme of silver nitrate is dissolved in 200 c.c. of 98 per cent. alcohol, 40 c.c. of ether, and 0.1 gramme of nitric acid added; (2) 15 c.c. of colza-oil dissolved in 100 c.c. of amyl alcohol. For a test, 5 c.c. each of the fat and solution No. 2, and 0.5 c.c. of No. 1 are shaken together and heated in a boiling water-bath for 15 minutes, the coloration being observed over a sheet of white paper. Should the indication appear doubtful, the fat may be pressed gently at 26° to 30°C. and both the oil and the cake examined separately.

F. H. L.

The Detection and Estimation of Glycerin in Beer. M. Molhant. (*Bull. de l'Assoc. Belge des Chim.*, 1895, pp. 17, 18.)—Glycerin is found in beer in amounts varying from 0·1 to 0·5 gramme per 100 c.c. The detection of as much as 0·6 gramme would, therefore, point to the artificial addition of glycerin.

In most of the methods employed the beer is evaporated in *vacuo*, and the residue taken up with various solvents, but the results are usually too low in consequence of mechanical loss during the manipulation.

With the following modified process of Clausnitzer the author has obtained very satisfactory results: 100 c.c. of the beer are evaporated in a porcelain dish on the water-bath to a syrupy consistence. Three grammes of slaked lime and about 8 grammes of pure calcined silica, or 10 grammes of quartz sand, are added. The mass is well mixed and then completely dried, finishing at 100° to 105° C., after which it is made into a cartridge, and extracted in a Soxhlet with 94 per cent. alcohol for at least six hours. When finished, the alcoholic extract is evaporated to about 50 c.c. and then cooled. The albuminoid bodies, dextrins, etc., are then precipitated by the addition of 30 to 40 c.c. of ether, while the glycerin remains in solution. The liquid is filtered into a weighed flask, and the filter washed with a mixture of alcohol and ether (2 : 3). The filtered liquid is evaporated, and the residue of glycerin dried at 100° to 105° until the weight is constant. The following results were obtained by this method:

Initial Beer.	Glycerin. Grammes.	Anhydrous Glycerin added in Grammes per 100 c.c.	Found. Grammes.
Mons Beer ...	0·21	0·50	0·70
"	0·21	1·00	1·18
Another Sample ...	0·18	0·50	0·68
"	0·18	1·00	1·14
Hal Beer ...	0·39	0·50	0·86
"	0·39	1·00	1·38
Another Sample ...	0·41	0·50	0·90
"	0·41	1·00	1·38

C. A. M.

Estimation of Boric Acid. H. Jay and Dupasquir. (*Comptes Rendus*, cxxi., 1895, pp. 260-262.)—The dried pulverized substance, which must be freed from organic matter, is made very slightly acid with hydrochloric or sulphuric acid, and introduced, together with 25 c.c. to 30 c.c. of methyl alcohol, into a flask fitted with a cork having two apertures. Through one of these passes a vertical tube slightly bent at its lower extremity, which reaches nearly to the bottom of the flask, and is connected with a condenser at the top. The other opening is for a tube which passes to the bottom of a second flask, the outlet of which is connected with the condenser of the first flask. One, two, or three c.c. of a normal solution of potash or soda, free from carbonic acid, are placed in the second flask, the amount being determined by the probable quantity of boric acid, and care being taken to have an excess. The two flasks connected together are then separately heated on the water-bath. The methyl

alcohol removes the boric acid from the first to the second flask, where it is retained by the alkali, while the methyl alcohol passes on through the condenser and back into the first flask, thus making a continuous extraction. The time occupied by the estimation varies, but does not as a rule exceed one and a half hours for a quantity of 300 milligrammes.

The alkaline liquid containing the boric acid is gently warmed to remove methyl alcohol, made up to definite volume, rendered slightly acid with several drops of dilute hydrochloric acid, and very gently warmed to volatilize any traces of carbonic acid. It is then titrated with decinormal potash or soda until turmeric paper indicates neutrality. The indicator "*blue C.L.B.*" is then added, and the titration continued to the neutral point again.

The conditions to be observed are always to work at constant temperatures, and to eliminate methyl alcohol and carbonic acid.

The following samples are given :

				Boric Acid Found. Grammes per Litre.
1 and 2.	Wines taken as typical	0.024
3.	Wine + 0.036 gramme HCl per litre	0.024
4.	" + 0.055 "	sodium fluoride	...	0.0255
5.	" + 0.100 "	sodium fluosilicate	...	0.0245
6.	" + 0.0062 "	boric acid	...	0.0289
7.	{ "	+ 0.124 "	boric acid	...
		+ 0.110 "	ammonium fluoride	...
8.	{ "	+ 0.100 "	boric acid	...
		+ 0.100 "	calcium fluoride	...
9.	{ "	+ 0.024 "	boric acid	...
		+ 0.072 "	sodium fluoride	...
10.	{ "	+ 0.055 "	boric acid	...
		+ 0.072 "	hydrochloric acid	...

It thus appears that fluoric acid causes an increase in weight, but in practice this slight error may be neglected, and the figure obtained attributed to boric acid alone.

Some figures as to the quantity of boric acid in certain animal and vegetable products are also given. Bordeaux and Burgundy wines (1891 and 1892) yielded amounts varying from 0.0105 to 0.022 gramme per litre. Three samples of cider and perry prepared in the laboratory gave 0.011 to 0.017 gramme per litre. The boric acid in four different wines varied from 0.008 to 0.017 gramme per litre. It was not found in a sample of beef-flesh examined by the author.

C. A. M.

Acetic Acid in Vinegar. A. R. Leeds. (*Jour. Amer. Chem. Soc.*, xvii., 1895, 741-744).—In most cases satisfactory results can be obtained by titrating 5 c.c. of the vinegar diluted to about 50 c.c. with seminormal alkali, using phenol-phthalein as the indicator. With highly-coloured vinegars, however, the end reaction is not sharp. The substitution of litmus-paper or solution as the indicator effects no improvement, lower readings being obtained, and these being lower by a variable amount in different vinegars.

The method of C. Mohr, recommended by Sutton and Wynter Blyth, of adding

an excess of pure calcium carbonate to a known quantity of vinegar and titrating the residual carbonate, failed utterly in the author's hands. To 50 c.c. of vinegar $2\frac{1}{2}$ grammes of finely-powdered calcium carbonate were added, the flask loosely corked, and shaken at intervals for six days. At the end of this time the contents were still acid. In a second experiment the flask was gently heated at intervals—some five or six hours in all. After filtering off the calcium carbonate, the residual acetic acid amounted to nine-tenths per cent. The figure calculated from the residual carbonate corresponded to 3.64 per cent., as against 4.44 per cent. found directly with soda. When a smaller amount of the vinegar (10 c.c.) was treated with an excess of the carbonate under a reflux condenser for two hours, a somewhat better result was obtained, the figure calculated from the residual carbonate being 3.85 per cent., while the liquid contained 0.72 per cent. acetic acid.

The author next tried the distillation process, in which 100 c.c. of 110 c.c. are distilled, and the distillate titrated, or the specific gravity taken. The distillate should contain 80 per cent. of the entire acid present. A trial by this method yielded a distillate with a specific gravity of 1.055 at 15° C., corresponding according to the tables to 4.25 per cent. By titration, the distillate showed (1) 4.23 and (2) 4.24 per cent., as against the correct percentage of 4.43 per cent., which figures correspond to 96 and not 80 per cent. of the total acid.

The process was then varied by distilling 10 c.c. of vinegar with 50 c.c. of water until 2 cc. remained in the retort, when another 50 c.c. of water was added, this being repeated twice more. Even after this long process only 4.39 per cent., or 99 per cent. of the total acid, was obtained. The distillate darkened on adding silver nitrate, and on standing gave a black precipitate.

The distillation was repeated in the same manner, using 10 c.c. of vinegar previously fortified with phosphoric acid. One trial gave 4.507 per cent. acid, another 4.514. The author suggests that the high figure was due to the presence of a little acetate.

Finally, to avoid the influence of the colouring matters, the following method was adopted: 50 c.c. of the vinegar, with 50 c.c. of water and a drop of phenolphthalein, were titrated with decinormal baryta, the latter being added to about 3 c.c. in excess, and this excess subsequently titrated back with decinormal sulphuric acid. The colouring matters subsided readily, either in the cold or upon warming. In one trial the precipitate was filtered off and washed before titrating back with acid, with a result of 4.48 per cent. In another the liquid was made up to 100 c.c., and 25 c.c. pipetted off and titrated; this gave 4.52 per cent. By using turmeric-paper as the indicator, the percentage was found to be 4.43 per cent., which was probably the correct amount.

C. A. M.

The Detection of Martius' Yellow in Macaroni, etc. F. Schaffer. (*Schweiz. Wochensh. Chem. Pharm.*, 1895, xxxiii., 251; through *Chem. Zeit. Rep.*, 1895, 216.)
—When 10-20 grammes of the macaroni or similar substance in small pieces are warmed and shaken with 40 c.c. of 50-60 per cent. alcohol, the presence of colouring matter is shown by the yellowing of the spirit. On the addition of a few drops of

hydrochloric acid, if saffron has been used, the colour is unchanged; if Martius' yellow, it disappears; if metanil yellow, it is changed to red. To determine whether Martius' or naphthol yellow S. has been employed, at least 200 grammes of the substance must be taken, and the alcoholic extract concentrated. Hydrochloric acid then gives with the former a white flocculent precipitate, soluble with a yellow colour in ether. With naphthol yellow S., hydrochloric acid produces no precipitate; but, even in the dilute solution, a flocculent precipitate is formed by caustic soda.

F. H. L.

The Determination of Starch. H. Ost. (*Chem. Zeit.*, 1895, xix., 1501.)—Various contradictory statements having been published by different observers regarding the feasibility of a quantitative estimation of starch, the subject has been carefully reinvestigated. The material employed was potato-starch, which had been repeatedly washed, freed from lumps, and finally air-dried. For the estimation of water, 1 to 5 grammes of the sample are heated in a Liebig's tube in a current of dry hydrogen, the temperature being maintained at 50° to 60° C. for about seven hours, then gradually raised to 120°. Working in this manner the results are very exact, while simple exposure to the air in a watch-glass placed in a drying-oven—the heating being arranged as before—gives figures only 0.1 to 0.3 per cent. too low. If, however, the temperature be raised too quickly, considerable hydrolysis may occur, even at 130° C., whereas a temperature of 150° will effect no injury if the drying be conducted slowly. (The process was checked by subsequent determination of the ash and ultimate analysis, and found perfectly trustworthy.)

For the estimation of the starch itself a number of processes were tried, the one which was found to answer best being that of Sachsse (*Chem. Centralbl.*, 1877, viii., 732), slightly modified. In this modification 3 grammes of the starch are heated with 200 c.c. of water and 20 c.c. of hydrochloric acid, specific gravity 1.125 (= 5.600 grammes of HCl), for two to three hours in a boiling water bath, using the factor 0.925 to calculate the glucose found in the starch. Longer heating gives results too low, and two hours on the water-bath are not sufficient. Slightly higher yields of glucose (89.8 instead of 89.5 per cent.) can be obtained by heating for a much longer period with less starch and acid, but there is no advantage to be gained by the alteration. Oxalic acid gives no better results. Dextrin may be determined in the same manner; also maltose, if 1 gramme of the latter be heated for five hours with 100 c.c. of 1 to 2 per cent. hydrochloric acid as before.

Of the optical methods of examination, the following modification of Effront's process (*Monit. Scient.*, 1887, 538) gives constant results, and may be used, therefore, for approximate commercial work; there is, however, decomposition of the starch: 2.5 grammes of the sample are rubbed down in a mortar, with 10 c.c. of hydrochloric acid (specific gravity 1.17) for eight to ten minutes, then diluted to 100 c.c., and polarized. The results varied 0.4°, from $[\alpha]_D = +196.3^\circ$ to 196.7° , for dry starch. The strength of the acid, and the time of rubbing together, must not be departed from.

By heating 2 to 3 grammes of the starch with 80 to 90 c.c. of plain water for three to five hours in a Lintner's flask to a pressure of 2 to 3 atmospheres, the use of the acid can be avoided. Solutions so obtained have no effect on Fehling's solution,

and, though somewhat opalescent, polarize well (+196.5 to 197.0). In cases where the energetic action of this process caused the solution to have a slight reducing action this was found to be without influence on the rotatory power. Baudry's salicylic acid method was not investigated.

The remainder of the paper, which is exceedingly interesting and of great length, is devoted to an examination of the products of the transformation of starch by diastase, glycerol, etc. The author comes to the conclusion that Lintner's isomaltose has no real existence, and that the series of bodies recently obtained by Zulkowski on heating starch with glycerol are not true dextrins, but compounds of those bodies with glycerol. He also expresses grave doubts as to the validity of the amyloïn theory of Brown and Morris.

F. H. L.

On the Action of Alkaline Copper Solutions on Sugars. J. Kjeldahl. (*Meddelelser fra Carlsberg Lab.*, V., 1895, pp. 1-63.)—It has often been noticed that in estimating sugar by the Soxhlet method, or one of its modifications, there has been but little agreement in the results obtained by different chemists. The author considers that much of this difference is due to the influence exercised by the atmospheric oxygen in increasing the weight of the cuprous oxide while heating the liquid. The action of oxygen during filtration need not be feared. That the manner in which the air has access to the vessel during heating influences the result was clearly shown by the following experiment :

I. 30 c.c. of Fehling's solution were added to a sugar solution containing 60 mg. of glucose in a 150 c.c. flask, and the liquid made up to 100 c.c. The stopper of the flask was fitted with two tubes, one reaching nearly to the bottom. Hydrogen was passed through for some minutes, the flask warmed on the water-bath, and the deposit filtered after twenty minutes through asbestos, dried, reduced in hydrogen, and weighed. Parallel determinations were also made in which the air had free access: II., in flask with narrow neck (100 c.c.); III., in conical flask, similar to that used in I.; IV., in a beaker; V., in a deep basin; VI., in a very shallow basin. The amount of surface in contact with the air is given in the first column below :

			Free Surface, square cm.	Copper found, mg.	Difference, mg.
I.	0	126.3	
II.	2	126.0	0.3
III.	17	123.9	2.4
IV.	21	121.5	4.8
V.	65	114.8	11.5
VI.	186	106.6	19.7

Concordant results were obtained on repetition, and it made no difference whether the liquid were cooled or not before filtration. It thus appears that to obtain satisfactory results the heating should be done in a current of hydrogen.

Experiments on glucose proved that within certain limits the proportion of caustic soda in the Fehling's solution had but little influence on the result. Thus, taking half or double the quantity only, showed a difference of 1 per cent. The

influence of the tartrate was somewhat greater, as doubling this constituent decreased the amount of reduced copper by 4 per cent. In the case of maltose and lactose the amount of soda used was of much greater importance.

However pure its constituents, Fehling's solution undergoes a slight reduction on prolonged heating, this being most marked in concentrated solutions. Thus, on heating for twenty minutes on the water-bath in a current of hydrogen, the following results were obtained :

100 c.c. Fehling's solution in 100 c.c.	...	11.3 mg. copper.
75 " " "	...	10.2 "
50 " " "	...	5.2 "
30 " " "	...	2.7 "
15 " " "	...	0.2 "

On prolonged warming this spontaneous reduction was greatly increased. Thus, 30 c.c. of Fehling's solution in 100 c.c., kept on a boiling water-bath for six hours, yielded 57 mg. of copper.

With regard to the length of time of boiling, the author found that after twenty minutes the weight of the copper increased only very slowly, and therefore fixed on that time as the limit for exposing to boiling water.

It will usually be found most suitable to use 30 c.c. or 50 c.c. of Fehling's solution in 100 c.c.; only for very small amounts of sugar is it necessary to use 15 c.c. in 100 c.c. From the examination of pure specimens of different kinds of sugar the author has constructed the following tables :*

15 C.C. FEHLING'S SOLUTION.

Copper, mg.	Glucose.	Fructose.	Invert Sugar.	Galactose.	Lactose, $C_{12}H_{22}O_{11} + H_2O$.	Maltose, $C_{12}H_{22}O_{11}$.
5	2.2	2.6	2.5	2.5	3.4	3.7
10	4.4	5.2	5.1	5.0	6.7	7.5
20	9.0	10.4	10.0	10.2	13.6	15.1
30	13.7	15.8	15.0	15.5	20.5	22.8
40	18.6	21.2	20.2	20.9	27.6	30.7
50	23.7	26.7	25.5	26.5	34.9	38.7
60	28.9	32.4	30.9	32.3	42.2	46.9
70	34.3	38.1	36.5	38.3	49.7	55.2
80	40.0	44.0	42.3	44.4	57.5	63.8
90	46.0	50.0	48.3	50.8	65.4	72.4
100	52.3	56.2	54.5	57.5	73.5	81.3
110	58.9	62.5	61.0	64.4	81.8	90.4
120	66.0	69.0	67.8	71.7	90.3	99.8
129	72.9	74.9	74.2	78.7	98.3	108.4

* The intermediate unit values have been omitted, to save space.—*Abst.*

30 C.C. FEHLING'S SOLUTION.

Copper, mg.	Glucose.	Fructose.	Invert Sugar.	Galactose.	Lactose, $C_{12}H_{22}O_{11} + H_2O$.	Maltose, $C_{12}H_{22}O_{11}$.
40	17.8	19.8	19.1	19.8	26.7	30.9
50	22.4	24.9	23.9	25.0	33.6	38.8
60	27.0	30.1	28.8	30.1	40.6	46.8
70	31.8	35.2	33.8	35.4	47.6	54.8
80	36.6	40.5	38.8	40.7	54.6	63.0
90	41.4	45.9	43.9	46.0	61.8	71.2
100	46.4	51.3	49.1	51.5	69.0	79.4
110	51.4	56.8	54.4	57.0	76.4	87.7
120	56.6	62.4	59.8	62.6	83.8	96.1
130	61.8	68.0	65.2	68.3	91.3	104.7
140	67.1	73.7	70.7	74.0	98.9	113.3
150	72.5	79.6	76.3	79.9	106.5	121.9
160	78.0	85.5	82.0	85.8	114.3	130.7
170	83.7	91.5	87.9	91.9	122.2	139.6
180	89.4	97.6	93.8	98.0	130.3	148.6
190	95.3	103.9	99.9	104.3	138.4	157.6
200	101.4	110.2	106.1	110.6	146.7	166.8
210	107.5	116.7	112.4	117.1	155.0	176.1
220	113.9	123.2	118.8	123.8	163.5	185.5
230	120.4	130.0	125.5	130.5	172.2	195.1
240	127.1	136.8	132.2	137.5	181.0	204.7
250	134.1	143.8	139.2	144.5	189.9	214.4
260	141.2	150.9	146.3	151.8	199.0	224.3

50 C.C. FEHLING'S SOLUTION.

Copper, mg.	Glucose.	Fructose.	Invert Sugar.	Galactose.	Lactose, $C_{12}H_{22}O_{11} + H_2O$.	Maltose, $C_{12}H_{22}O_{11}$.
100	44.7	49.1	47.2	49.5	71.4	82.9
110	49.4	54.3	52.2	54.6	78.7	91.3
120	54.1	59.4	57.1	59.8	86.1	99.8
130	58.8	64.6	62.0	65.0	93.5	108.4
140	63.7	69.8	67.0	70.3	100.9	117.0
150	68.5	75.2	72.1	75.6	108.4	125.7
160	73.4	80.5	77.2	81.0	115.9	134.3
170	78.4	85.9	82.4	86.4	123.0	143.0
180	83.4	91.3	87.6	91.9	130.9	151.8
190	88.4	96.8	92.9	97.4	138.5	160.6
200	93.5	102.3	98.2	102.9	146.1	169.4
210	98.7	107.9	103.6	108.5	153.8	178.3
220	103.9	113.5	109.0	114.2	161.5	187.2
230	109.2	119.2	114.5	119.9	169.3	196.2
240	114.5	124.9	120.0	125.7	177.1	205.2
250	119.9	130.7	125.6	131.5	184.9	214.3
260	125.4	136.5	131.2	137.4	192.7	223.3
270	131.0	142.4	137.0	143.3	200.6	232.5
280	136.6	148.3	142.7	149.3	208.6	241.7
290	142.3	154.3	148.6	155.4	216.6	251.0
300	148.0	160.4	154.5	161.5	224.6	260.2
310	153.9	166.6	160.5	167.7	232.7	269.6

Copper, mg.	Glucose.	Fructose.	Invert Sugar.	Galactose.	Lactose, $C_{12}H_{22}O_{11} + H_2O$.	Maltose, $C_{12}H_{22}O_{11}$.
320	159.8	172.8	166.6	174.0	240.8	279.0
330	165.8	179.1	172.7	180.3	249.0	288.4
340	171.9	185.5	179.0	186.8	257.2	297.9
350	178.1	191.9	185.3	193.3	265.4	307.5
360	184.4	198.4	191.7	199.8	273.7	317.0
370	190.8	205.0	198.2	206.5	282.1	326.7
380	197.3	211.7	204.8	213.3	290.5	336.4
390	204.0	218.5	211.5	220.1	299.0	346.2
400	210.7	225.3	218.3	227.1	307.5	356.0
410	217.6	232.3	225.2	234.1	316.0	365.9
420	224.6	239.4	232.3	241.2	324.6	375.8
430	231.8	246.6	239.5	248.5	333.3	385.8
434	234.7	249.5	242.4	251.5	336.8	389.9

To determine the equivalent of acid formed by oxidation of a molecule of sugar, a modification of the iodometric method may be used. This is based on the reactions :



The first reaction is only complete with strong inorganic acids, but by adding an excess of thiosulphate after the potassium iodide and iodate it is soon brought about. On the following day the excess of thiosulphate is titrated with a standard iodine solution. Care must be taken to ensure the absence of carbonic acid before adding the mixture of iodine and thiosulphate.

The author's experiments on glucose and fructose show that, besides formic acid, the oxidation products contain acids of the type $\text{C}_n\text{H}_{2n}\text{O}_{n+1}$, with glycolic, glyceric, trioxylbutyric, arabonic, and gluconic acids.

Some experiments on the action of hot sodium hydrate by itself on sugars point to the conclusion that one molecule (180) of glucose or fructose, or $\frac{n}{6}$ molecule of arabinose, give 1.63 acid equivalents; whereas the second group, comprising galactose, maltose, and lactose, give for 180 of sugar an acid equivalent of 1.45.

C. A. M.

The Volumetric Estimation of Sugar by means of Ammoniacal Copper Solution. Z. Peska. (*Rozprawy ceske Akademie*, 1895, v. (II.), No. 19; *nrough Chem. Zeit. Rep.*, 1895, 257.)—In order to avoid the oxidation of the copper suboxide in solution, the author now uses a layer of vaseline, instead of the usual current of hydrogen. Two solutions are prepared: 6.927 grammes of the purest crystallized copper sulphate are dissolved in water, 160 c.c. of 25 per cent. ammonia added, and the whole made up to 500 c.c.; 34.5 grammes of Rochelle salt and 10 grammes of caustic soda are also dissolved and diluted to 500 c.c. For the analysis, a mixture of 50 c.c. of each liquid is heated in a beaker under a layer of vaseline oil 5 mm. thick, to a temperature of 80° C. The sugar solution is run in 1 c.c. at a time for the first test, but on a repetition the whole amount may be added at once. Towards the end of the titration, the temperature must be raised to 85°, and the heating continued for

* $\bar{\text{A}}$ represents a monovalent acid residue.

two minutes when working on either glucose or invert sugar, four minutes for maltose, and six minutes for milk sugar. Dextrin increases the reducing power of the sugar in this solution less than in the one prepared with potash, and as the ammonia has no injurious action, the whole process is both exact and convenient. When saccharose is present, 1 gramme of it has a reducing action equivalent to 0.0026 gramme of invert sugar. In the determination of lactose in milk the albuminoids should be precipitated with lead acetate and the excess of lead removed by sodium sulphate. The following table gives directly the number of milligrammes of each sugar in 100 c.c. of solution.

C.c.'s used.	Glucose.	Invert sugar.	Milk sugar.	Maltose.	C.c.'s used.	Glucose.	Invert sugar.	Milk sugar.	Maltose.
8	997.8	1049.2	—	—	50	163.0	173.2	318.1	360.0
9	889.4	935.1	—	—	51	159.8	169.8	311.9	353.0
10	802.3	844.6	—	—	52	156.8	166.5	306.0	346.3
11	730.7	770.0	—	—	53	153.9	163.4	300.3	339.9
12	670.8	707.6	—	—	54	151.1	160.4	294.8	333.8
13	620.0	654.5	—	—	55	148.4	157.5	289.4	327.9
14	576.3	608.7	—	—	56	145.7	154.7	284.2	322.2
15	538.4	568.9	1033.9	—	57	143.1	152.0	279.3	316.7
16	505.2	534.2	971.4	—	58	140.6	149.4	274.5	311.4
17	475.8	503.3	916.0	1023.0	59	138.2	146.9	269.9	306.3
18	449.7	475.7	866.5	968.8	60	135.9	144.5	265.4	301.3
19	426.3	451.2	822.3	920.3	61	133.7	142.2	261.1	296.4
20	405.2	429.0	782.4	876.3	62	131.5	139.9	256.9	291.6
21	386.0	408.8	746.0	836.4	63	129.4	137.7	252.9	287.0
22	368.7	390.6	713.0	800.0	64	127.4	135.5	249.0	282.6
23	352.8	373.8	682.7	766.5	65	125.4	133.4	245.2	278.3
24	338.2	358.4	654.8	735.8	66	123.5	131.4	241.5	274.1
25	324.8	344.3	629.2	707.5	67	121.7	129.5	237.9	270.0
26	312.4	331.2	605.5	681.3	68	119.9	127.6	234.4	266.1
27	300.9	319.3	583.5	656.8	69	118.2	125.7	231.0	262.3
28	290.3	307.8	563.1	634.1	70	116.5	123.9	227.7	258.6
29	280.3	297.3	544.1	613.0	71	114.9	122.2	224.6	255.0
30	271.1	287.5	526.2	593.2	72	113.3	120.5	221.5	251.5
31	262.4	278.2	509.5	574.5	73	111.8	118.9	218.5	248.1
32	254.2	269.6	493.8	557.1	74	110.3	117.3	215.6	244.8
33	246.6	261.6	479.1	540.8	75	108.8	115.8	212.8	241.6
34	239.3	253.9	465.3	525.3	76	107.4	114.3	210.0	238.4
35	232.6	246.7	452.2	510.7	77	106.0	112.8	207.3	235.3
36	226.1	240.0	439.8	496.8	78	104.6	111.4	204.7	232.3
37	220.0	233.5	428.1	483.7	79	103.3	110.0	202.1	229.4
38	214.3	227.4	417.0	471.3	80	102.0	108.6	199.6	226.6
39	208.8	221.7	406.5	459.5	81	100.8	107.2	—	223.9
40	203.6	216.2	396.5	448.3	82	99.6	105.9	—	221.2
41	198.7	211.0	387.0	437.6	83	—	104.6	—	218.6
42	194.1	206.0	377.8	427.4	84	—	103.4	—	216.0
43	189.7	201.3	369.2	417.7	85	—	102.2	—	213.5
44	185.4	196.7	360.9	408.4	86	—	101.1	—	211.1
45	181.2	192.3	353.0	399.5	87	—	—	—	208.7
46	177.3	188.1	345.4	391.0	88	—	—	—	206.4
47	173.5	184.1	338.1	382.8	89	—	—	—	204.1
48	169.9	180.3	331.2	374.9	90	—	—	—	201.9
49	166.4	176.7	324.5	367.3	91	—	—	—	199.7

Preparation of Sugar Solutions for Polarimetry. Stift and Petziwal. (*Oesterr. Zeits. Zuckerind.*, 1895, xxiv., 487; through *Chem. Zeit. Rep.*, 1895, 239.)—The authors point out several objections to the use either of tannin or lead acetate for decolorizing sugar solutions. On the other hand, they strongly advise the employment of Herles' plan, which consists in the employment of lead nitrate. It works well, even in the case of very dark liquids. F. H. L.

The Influence of Lead Acetate on the Determination of Invert Sugar. Borntäger. (*D. Zuckerind.*, 1895, xx., 1169; through *Chem. Zeit. Rep.*, 1895, 239.)—The author confirms Gill's old statement that lead acetate causes the amount of sugar as found by Fehling's solution to be too low. The best method of removing the excess of lead salts, especially in the case of wine analysis, is by means of sodium phosphate. The experiments are being continued. F. H. L.

The Chemical Nature of Diastase. T. B. Osborne. (*Jour. Amer. Chem. Soc.*, xvii., 1895, pp. 587-603.)—The methods usually employed for the isolation of the enzymes have been to extract the enzyme-containing tissues with water or glycerol, the enzyme being subsequently precipitated in an impure condition from the extract by the addition of alcohol. An attempt was then made to free the enzyme from its attendant impurities by repeated solution in water and re-precipitation with alcohol, the mineral matters being removed by dialysis. According to the author, these processes, the former of which was used by Lintner for the preparation of diastase, are not calculated to yield pure preparations, as the precipitate produced by alcohol contains nearly all the proteid matter present in the extract, as well as a large amount of carbohydrates and salts.

A better method is to first precipitate the proteids, together with the diastase, by saturating the extract with ammonium sulphate, next to remove the globulins by dialysis and, finally, to separate the albumin and proteoses by dialysis in alcohol. The ammonium sulphate does not exercise any injurious action on the diastase. Lintner recommended extraction of the malt with 20 per cent. alcohol, but the author found that although preparations with a high diastatic power could be thus obtained, the method is not so suitable for subsequent precipitation with ammonium sulphate as extraction with water.

Ten kilogrammes of malt were exhausted with water, and the extract saturated with neutral ammonium sulphate. The precipitate was dialyzed in 4 litres of water, until much of the sulphate had been removed, and the precipitated proteid largely dissolved. The insoluble residue, consisting largely of globulin, was then filtered off, and the filtrate saturated with ammonium sulphate. This precipitate was also dialyzed in 1,500 c.c. of water. By this treatment most of the globulin present in the extract was separated, and, after filtration, the clear filtrate was dialyzed into an equal volume of alcohol (specific gravity 0.84). After twenty-four hours precipitate I. had formed and was filtered off. The filtrate was again dialyzed into alcohol of the same strength, and after twenty-four hours precipitate II. was obtained. On dialyzing the filtrate into somewhat stronger alcohol, precipitate III. separated out, while precipitate IV. was

obtained in a similar manner. The filtrate from this yielded precipitate V. on the addition a large quantity of absolute alcohol. In this way all the proteid in the extract was separated. The approximate weights of the precipitates were: I., 13; II., 8; III., 6; IV., 5; and V., 3 grammes.

Precipitate I. was largely insoluble in water, and, after filtration and dialysis of the filtrate, yielded a preparation with a diastatic power of 30 by Lintner's test. It was found to consist largely of proteose. The portion insoluble in water consisted of globulin-like substances.

Precipitate II. was treated with water, and the solution dialyzed for several days in water, a quantity of absolute alcohol being finally added, which precipitated the proteid. The dried preparation was almost completely soluble in water, and the solution on heating became turbid at 60° C., and flocculent at 66° C. It contained a slight amount of insoluble matter, some albumin, and much proteose. Its diastatic power was 75. The part insoluble in water was similar in composition to the corresponding residue in I.

Precipitate II. contained less globulin and proportionately more albumin and proteose than I., and had, therefore, a higher diastatic power.

Precipitate III. was treated in the same way as II. The resulting precipitate, which was almost completely soluble in water, yielded a solution which became turbid at 55° and flocculent at 60°. The diastatic power of the preparation was 122. The insoluble residue was considered to be impure globulin.

Precipitate IV., treated as in the other cases, yielded a preparation which dissolved to a nearly clear solution in water. The filtered solution became turbid at 50° and coagulated at 56°. This preparation had a diastatic power of 600. At 20° it was able to produce from soluble starch 2,000 times its weight of maltose. Its composition, neglecting ash, was: Carbon, 52.50; hydrogen, 6.72; nitrogen, 16.10; sulphur, 1.90; oxygen, 22.78. This diastase had therefore six times the diastatic power of the most active preparation made by Lintner; which, since it only contained 10.42 per cent. nitrogen, led him to conclude that diastase was not a true proteid.

Precipitate V. dissolved completely on treatment with water. It was precipitated and washed with absolute alcohol, and yielded a preparation with a diastatic power of 60. Its aqueous solution became turbid at 50° and flocculent at 58°. It consisted chiefly of proteose.

It thus appears that in malt extract there is a globulin, an albumin, and at least one (more probably two) forms of proteose. The amount of proteose diminished from precipitate I. to IV., which contained the least, while V. was mainly proteose. The globulin is rendered insoluble more readily than the albumin, and may thus be separated.

The author's general conclusion is that diastase is most closely related to the albumin, this being shown by the behaviour and composition of the preparation obtained from precipitate IV. The amount of coagulable albumin in this preparation was found to be 53.2 per cent. of the dried substance.

W. J. S. & C. A. M.

NOTE BY THE ABSTRACTORS.—We are able to confirm the statement that the

activity of malt diastase is not affected by precipitation with ammonium sulphate. Acting upon a hint given in the original paper, we find that the diastase is completely precipitated from aqueous malt extract on saturation with magnesium sulphate, without its activity being diminished. The precipitate obtained with the latter salt is infinitely smaller in bulk than that obtained by means of the former.

The Detection of Water in Acetone. H. Schweitzer and E. E. Lungwitz. (*Chem. Zeit.*, 1895, xix., 1384.)—By shaking together equal volumes of acetone and petroleum ether (b. p. 40 to 60° C.) the presence of a small quantity of water in the former is shown by the separation of the liquid into two layers. With dry acetone no such separation takes place. The reaction is only qualitative. By the employment of a heavier spirit (b. p. 80 to 100° C.), followed by distillation, the authors are attempting to work out a process for commercial use. F. H. L.

Antipyrine and the Thalleioquin Reaction. J. Ducommun. (*Schweiz. Wochensch. Chem. Pharm.*, 1895, xxxiii., 242; through *Chem. Zeit. Rep.*, 1895, 214.)—One part of antipyrine in the presence of twenty parts of quinine prevents the appearance of the well-known green colour of this test, producing in its stead a fine red. Urea stops the production of either colour, while the salts of morphine, pilocarpine, cocaine, strychnine, codeine and atropine, chloral hydrate, and phenol, etc., in 1 per cent. solutions are without influence on the green tint. F. H. L.

The Estimation of Iodine in Organic Substances of the Fatty Series. M. C. Schuyten. (*Chem. Zeit.*, 1895, xix., 1143.)—A weighed amount of the substance, chosen so as to contain 0.03 to 0.05 gramme of iodine, is put into a glass tube holding about 15 c.c., then some freshly-melted and finely-powdered potassium bichromate mixed with it by agitation, and finally a layer 5 to 6 c.m. thick of bichromate. The end of the tube is drawn out and bent. By gentle heating, the iodine is then sublimed into the narrow part of the tube, which is kept cold by a wet cloth. When all has come off, and the molten substance is perfectly clear, the tube is cut off, the iodine washed out with potassium iodide solution, and titrated in the usual manner. It may also be weighed direct by placing the cut-off tube in connection with a tube containing calcium chloride and some lumps of caustic soda till all moisture has disappeared. The results quoted by the author are satisfactory. F. H. L.

Ionone and Irone. F. Tiemann and P. Krüger. (*Ber.*, 1895, xxviii., 1754.)—The authors describe several condensation products of these ketones, by means of which their presence and purity may be established. The semicarbazides of both substances are produced by the action of semicarbazide sulphate on the ketones in glacial acetic acid solution, the ionone compound melting at 109° to 110° C. From a cold solution of parabromophenylhydrazine in acetic acid of such a strength that it will bear the addition of an equal or double volume of water without crystallizing,

ionone - parabromophenylhydrazone may be prepared as a white or light-yellow crystalline precipitate. If the ionone is pure, the hydrazone appears in very characteristic glittering leaf-like crystals, which, when dry, soften at 134°, and melt to a clear oil at 140° to 145° C., crystallizing again on cooling. Impure preparations may be recrystallized from hot methyl alcohol to which a little water has been added. Mineral acids gradually convert the hydrazone into the original ionone, as may be detected by the violet-like odour.

The corresponding compound of irone may be prepared in a similar manner; it crystallizes in groups of needles, which soften at 156°, and melt at 168° to 170° C. When water is added gradually to a cold or slightly warm acetic acid solution of the two hydrazones, the irone compound is first precipitated, and the ketones may be separated in this manner. The above-described reactions take place quantitatively, and they can be employed for the estimation of either perfume. F. H. L.

Volumetric Estimation of Arsenic in Iron and Steel. A. Mignot. (*Rev. Chim. anal. appliq.*, 1895, iii., 101; through *Chem. Zeit. Rep.*, 1895, 164.)—Ten grammes of the metal are dissolved in nitric acid and evaporated, and the residue ignited to decompose the nitrates; the powdered mass is treated with concentrated hydrochloric acid in the cold for an hour. The whole, solution and residue, is introduced into a long-necked, 500 c.c. flask, made up to 300 c.c. with strong hydrochloric acid, and 50 c.c. of saturated ferrous chloride solution added. This flask is connected, through a condenser, with a small flask, into which the leading-tube only just enters, while a second tube, starting from the bottom of the latter, is bent so as to dip into some water contained in a beaker. The distillation is continued till only 100 c.c. are left in the first flask, care being taken, by removing periodically the beaker, that the distillation does not become sufficiently violent to cause any of the liquid to boil over. The distillate is made up to 500 c.c., precipitated with sulphuretted hydrogen, and the sulphide filtered off, washed and dissolved in 20 c.c. of (1:3) ammonia. It is again precipitated with 20 c.c. of acetic acid of $\frac{5}{4}$ times the equivalent strength, iodine solution added, and the excess titrated with thiosulphate. The iodine is standardized on an arsenic solution precipitated, etc., as above described. It is stated that this process will estimate 0.005 grammes of arsenic. (Compare Fresenius, *Quant. Anal.*, vol. ii., 6th (German) edition, p. 558 *et seq.*)

F. H. L.

The Separation of Arsenic from other Elements by means of Methyl Alcohol and Hydrochloric Acid. C. Friedheim and P. Michaelis. (*Ber.*, 1895, xxviii., 1414.)—Fischer's method for the estimation of arsenic by distillation with ferrous chloride and hydrochloric acid is inconvenient in many cases, as the presence of the ferric chloride resulting from the operation complicates the determination of other substances in the liquid after the arsenic has been removed. Methyl alcohol is free from this disadvantage, as it adds nothing to the bodies under treatment beyond a small amount of carbon.

The liquid is introduced into a 250 c.c. flask fitted with a stoppered dropping

funnel, and connected with a condenser and a flask of 750 c.c. capacity, in which 20 c.c. of strong nitric acid are placed. To this flask is connected a set of potash bulbs filled with water, all the joints being preferably ground in. To the liquid are added 50 c.c. of methyl alcohol, which must be kept as dry as possible, and if much water has been employed in introducing the substance into the flask, most of this should be first distilled off. A rapid stream of dry hydrochloric acid gas is introduced through the funnel, the flask being kept cold, and care being exercised that the liquid is not drawn back. When completely saturated, the stream of gas is slackened, a water-bath attached, and the whole distilled. According to the amount of arsenic and water present, the operation is repeated a second or third time, more alcohol being added if required.

The distillate, etc., is washed out into a large porcelain basin, 20 to 30 c.c. of strong nitric acid added—the basin being covered over to avoid loss during the evolution of gas—and then the liquid evaporated down to 100 c.c. A second equal amount of nitric acid is employed, and the whole evaporated to dryness, taken up in water, and precipitated with magnesia mixture. The authors have satisfactorily effected the separation of arsenic from vanadium, tungsten, and molybdenum by this process.

F. H. L.

The Preparation of Pure Zinc. F. Mylius and O. Fromm. (*Zeits. Anorgan. Chem.*, 1895, ix., 144; through *Chem. Zeit. Rep.*, 1895, 198.)—By the term "pure" zinc the authors understand a metal spectroscopically as well as chemically pure, whereas the purest commercial zinc usually contains iron, lead, and cadmium in amounts of at least 1.4, 5, and 16 parts per 100,000 respectively. The latter two impurities are best detected and estimated by fractional precipitation with ammonium sulphide in an ammoniacal nitrate solution, the reagent being added until the precipitate falls of a pure white colour. For the preparation of the pure metal fractional crystallization of commercial zinc is useless, but by boiling the nitrate solution freed from other metals as indicated and igniting the precipitate, pure zinc oxide may be obtained. By electrolyzing a solution of the sulphate, using zinc oxide to neutralize the acid set free, a product is obtained contaminated with platinum from the anode. The purest metal may be prepared by the repeated electrolysis of a basic sulphate solution, the zinc being finally sublimed *in vacuo*, when it will be found not to contain more than 1 per 100,000 parts of impurity.

F. H. L.

The Volumetric Determination of Zinc, and a New Indicator for Ferrocyanide. G. C. Stone. (*Journ. Amer. Chem. Soc.*, 1895, xvii., 473-477.)—In titrating zinc with ferrocyanide, the metals of the iron group must first be removed. Since no rapid and accurate method for separating manganese and zinc is published, the author proposes titrating them together, determining the manganese in a separate portion by titration with permanganate, and taking the zinc by difference. The best indicator for ferrocyanide when used with manganese was found to be cobalt nitrate. A drop of a quite dilute solution is placed on a tile beside a drop of the solution to

be tested, just touching but not mixing with it. The end reaction is shown by an immediate faint green line at the junction of the drops.

The best strength for the ferrocyanide solution is about 30 grammes per litre. It is standardized by titrating solutions containing known amounts of zinc or manganese, making slightly acid with HCl, and keeping the solution at about the volume used in the analysis. The amount of ferrocyanide necessary to give a reaction with cobalt in this volume of acidified water must also be determined, and the result deducted for each titration.

The permanganate solution is standardized in the usual way with iron, the result being multiplied by 0.294646. It contains 1.99 grammes of crystallized potassium permanganate per litre, 1 c.c. being equivalent to 0.001 gramme of manganese. The ore is dissolved in HCl with the addition of KClO_3 as an oxidizer, and care must be taken to have sufficient acid to keep all the manganese in solution.

Lead alone need not be separated; copper can be precipitated by lead; or lead and copper can both be precipitated by aluminium. Cadmium should be precipitated by H_2S , and the filtrate oxidized. Iron and aluminium are best separated by barium carbonate, but the latter must be free from alkaline carbonates and hydroxides, barium hydroxide and ammonium salts. A salt sufficiently pure for the purpose may be obtained by suspending the ordinary "pure" carbonate (first proved free from ammonium salts) in warm water for several hours with 2 or 3 per cent. of its weight of barium chloride.

The well-oxidized solution of the ore is put into a 500 c.c. flask, and barium carbonate suspended in water added until the precipitate coagulates. The whole is then poured into a beaker, well mixed, allowed to settle, and the clear liquid decanted through a dry filter. Portions of 50, 100, or 200 c.c. of the filtrate are used for each titration. One portion, which should contain between 0.01 and 0.04 gramme of manganese, is diluted to 200 c.c., heated nearly to boiling in a porcelain dish, and titrated rapidly with permanganate with vigorous stirring.

In a second portion made *slightly* acid with hydrochloric acid, the zinc and manganese are titrated together in the cold with ferrocyanide; the dark colour of the precipitate suddenly changes to light yellowish green shortly before the end of the reaction. It is not necessary to test with the cobalt solution until 1 or 2 c.c. of the ferrocyanide solution have been added after the lightening of the precipitate.

To show the calculation of the results, the following example is given: 1 c.c. of the ferrocyanide solution equalled 0.00606 gramme of zinc, or 0.00384 of manganese; 1 c.c. of the permanganate equalled 0.001 gramme of manganese. $2\frac{1}{2}$ grammes of the ore were dissolved, and the iron precipitated and filtered out. 50 c.c. of the solution were diluted, heated, and titrated with permanganate, requiring 18.45 c.c. = 7.38 per cent. of manganese. 100 c.c. titrated with ferrocyanide required 27.85 c.c., of which 9.61 c.c. would be used by the manganese present. Deducting this, 18.24 c.c. was left for the zinc, equal to 0.11053 gramme, or 22.11 per cent. The amounts of zinc and manganese as determined gravimetrically were 22.05 and 7.58 per cent. respectively. The other results tabulated are equally satisfactory.

C. A. M.

An Improved Burette. F. Oettel. (*Chem. Zeit.*, 1895, xix., 1384.)—The upper end of an ordinary Mohr's burette is expanded to form a funnel of about 30 mm. diameter, which may be closed by a sphere of glass to prevent entry of dust. So improved, the burette may be slung from this funnel, and tilted out of the vertical when titrating hot solutions. The graduations are not obscured by the ordinary clamp, and filling is much facilitated.

F. H. L.

REVIEWS.

THE SCIENCE AND ART OF BREAD-MAKING. By WILLIAM JAGO, F.I.C. (London: Simpkin, Marshall, Hamilton and Co.) Price 15s. net.

It is most difficult to form an idea for what class of reader this bulky volume has been compiled, whether for the scientist or for the baker and miller. If for the former, it contains a great deal of matter altogether unnecessary, and in many instances of doubtful value; if for the latter, it is highly questionable if there are many millers or bakers in the country who will be any the wiser for reading it.

That part of the work which relates more strictly to bread making and baking, and which contains much original work carried out by the author, is, though diffuse and often smacking of objectionable self-advertisement, a valuable contribution to the knowledge on the subject. The work of C. O'Sullivan, Horace Brown, and other investigators on the carbohydrates is fairly and clearly given; so also are the modern views as to the nature of fermentation. Analytical processes for the examination of flours and bread are also given in great detail and with accuracy.

The scientific introductory chapters, on the other hand, are open to much criticism, and contain very numerous statements and expressions to which exception may be taken. The first three chapters, which travel over the whole region of chemistry and physics, bristle with inaccuracies and slipshod writing. We quote a few of these: "Many, if not most, liquids mix readily with water in all proportions"; "Most solid bodies dissolve in water"; "Phosphorus occurs ordinarily in sticks"; "Calcium is scarcely known in the free state"; "Calcium hydrate occurs as a dry white powder"; "It has been proposed to define organic chemistry as the chemistry of carbon compounds." This definition, however, does not please the author, so he defines organic chemistry "as that branch of the science which treats of the composition and properties of those compounds whose usual source is either animal or vegetable." Under this definition, therefore, all such compounds as zinc-ethyl would be removed from the realm of organic chemistry! "The temperature of the boiling-points increases," etc.; "The alcohols are hydrates of the organic radicals." Mannite is stated to be the mother substance from which all the other carbohydrates are supposed to be derived—a statement which is only true for some of the hexoses. Margaric acid is still quoted as a constituent of natural fats; oleic acid is stated to be the product of oxidation of an alcohol; "Oleates of glycerin constitute the oils"; "The separation of fats into glycerin and the fatty acids may be effected by forcing a current of steam through the melted fat"; "But little is understood of the constitution of the alkaloids." And so on *ad infinitum*.

The chapter on the constitution of the carbohydrates is a long way behind the present state of science. The author, for instance, says that the glucoses (dextrose, lævulose, and galactose being the only three representatives referred to by him) are the aldehydes of mannite. It is now well known that lævulose is not an aldehyde, but a ketose. Vegetable ivory is stated to be nearly pure cellulose; it contains, however, a large quantity of mannan, the parent substance of mannose. Lævulose is stated to be not crystallizable, although crystallized lævulose has been for some years an article of commerce. An analysis by the author of the fat of the germ of wheat shows 26.93 per cent. of glycerin, and the analytical results sum up to 100, although the glycerin and fatty acids are given as such without subtraction of water. This percentage of glycerin cannot be accepted as even an approximation to the truth.

A most objectionable feature of the book, from a professional point of view, is the advertising part, both without and within the body of the work. There are forty-two pages of advertisements, exactly one-third of which contain the name of the author, either in advertising reports given to makers of all sorts of articles, or in direct advertisements on behalf of Mr. Jago. A long list of the names and addresses of millers whose flours are described in the body of this work is also given, and copies of certificate-forms, with Mr. Jago's name and qualifications, are contained in the text of the book itself. This is hardly consonant with the present standard of professional ethics.

Altogether the work is of most unequal merit, for the sound information contained in the book is in danger of being stultified by its numerous inaccuracies.

O. H.

POISONS, THEIR EFFECTS AND DETECTION. By A. WYNTER BLYTH. (London: C. Griffin and Co. Price 21s.)

This work is undoubtedly the most complete treatise on toxicology in our language, and should be in the library of every analyst and teacher interested in the subject. It begins with a brief but very interesting historical sketch, carrying us in twelve pages from Greek myths, through ancient Egyptian and Hebrew lore, down to the Italian poisoners of the seventeenth century.

This is followed by some pages of an equally interesting account of the improvements introduced from time to time into our methods for the detection of poisons. These chapters will be highly appreciated by all earnest students who, not having time or opportunity to study the original memoirs, have yet the wish to possess some knowledge on these subjects. We heartily thank the author for the trouble he has taken to bring these facts to our knowledge in so compendious a form.

Next follow chapters common to all works on the subject—the definition of a poison and the classification of poisons; but even on these the author has succeeded in impressing his own individuality. The author does not, however, follow the beaten track for long, but next favours us with several chapters on the statistics of poisoning in England, Germany, and France, and a very valuable account on “the connection between toxic action and chemical composition.” In these chapters the author breaks practically new ground, as far as general works on toxicology are concerned, and all students will be indebted to him for so clearly and concisely bringing together

the scattered information on this important subject. With a series of chapters on the identification of poisons by experiments on animals, a general method in searching for poisons, the use of the spectroscope, and the examination of blood-stains, what we may term the introductory part of the work comes to an end. It will be seen in how unusually comprehensive and complete a manner this portion of the subject has been treated, and our gratitude is due to the author for the able way in which he has carried out his plan.

Where so much is given it may appear ungracious to ask for more, but we cannot help expressing a wish that in future editions the author will favour us with some general remarks on the following subjects, which are of great importance to the practical toxicologist, but are greatly neglected in all works on toxicology—namely, some account of the rate of absorption of poisons in various forms and in various ways, together with their distribution during absorption in the various organs and tissues of the body; the rate at which poisons are either eliminated from, or destroyed within, the system, and the relative proportions in which they are found in the various organs and tissues, etc., in the course of such elimination. We know no one who could better give us information on these subjects than the gifted author of the work before us. It is impossible, in the space at our command, to give anything like a full account of the contents of this handsome volume of over 700 pages, and we must content ourselves with a few general remarks regarding the remainder of the volume.

In the first place, the author has departed somewhat from the usual order in treating the various poisons. This departure is based on the author's general method for the detection of poisons, and is fully justified thereby. Ample information is generally given regarding the physiological and chemical characters of the various poisons treated of, together with the best methods for their separation and identification. This part of the work is very complete; in fact, the details given are sometimes too ample, and render the work, occasionally, rather difficult as a practical guide. We have found the information given generally reliable and fully up to date. In some places, however, the author adopts what we cannot but call the inconvenient plan of referring the reader to other works of his for further information, instead of, as ought to have been done, giving the information in the present volume. Some readers may also regret the almost total absence of records of important trials, which render Taylor's great work so interesting and instructive.

In conclusion, we can heartily recommend the work to all our readers, and congratulate the author on the extremely able and original manner in which he has accomplished a task of no common difficulty.

A. D.

AIR, WATER, AND DISINFECTANTS. By C. M. AIKMAN, M.A., D.Sc., F.R.S.E.
(Society for Promoting Christian Knowledge.) Price 1s.

This handy little book, which forms one of a series of "Manuals of Health" published by the above society, contains much useful information put into exceedingly readable form. The instruction imparted is trustworthy and well up to date. The work cannot fail to be of considerable use to the class of readers to which it is addressed.

THE ANALYST.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

FREE FATTY ACIDS IN OIL-CAKES AND OTHER FEEDING STUFFS.

BY BERNARD DYER, D.Sc., AND J. F. H. GILBARD.

(Read at the Meeting June 5, 1895.)

IN January, 1893, we contributed to the *Journal of the Society of Chemical Industry* some notes on this subject, giving the results of the examination of the oil in 100 samples of linseed cake. The investigation was subsequently extended to the examination of nearly 1,000 cakes and feeding stuffs of various kinds, taken just as they happened to be sent for analysis. We were not the first to call attention to the fact that oil-cakes—especially those in bad condition, or made from heated seed—frequently contained a considerable proportion of free fatty acid; but not many determinations had, as far as we had seen, been published, and as the titration of the oil extracted in the ordinary course of analysis is a simple operation, it appeared a sufficiently interesting matter to warrant, for a time, the expenditure of the small trouble necessary, even if the results should turn out to have no further use than the gratification of our own curiosity. Our accumulated notes on the subject are now placed on record for the information of others who may feel interested in the matter.

We pointed out before that many circumstances might be suggested as influencing the production of free fatty acid. The most notable is the fermentation, or "heating," either of the seed before the crushing operation, or subsequently of the cake itself, owing to its being too moist, or to its being badly stored, either on shipboard, or in the warehouse, or in the farmer's barn. Furthermore, it seemed not impossible that the steaming of the ground seed before crushing might sometimes result in some degree of decomposition of the oil; while, on the other hand, such heating might perhaps tend to "sterilize" the cake, and so check subsequent possible bacteriological decompositions which might involve alterations in the oil.

As a matter of fact, in cakes which we have had reason to know to be newly made, we have found, as a rule, a very low degree of free acidity. It will be seen, for instance, that in 116 freshly-made cakes only about 3 per cent. of the oil was free acid, and in more than 100 of these samples the proportion did not exceed 5 per cent. of the oil. In 11 samples it was between 5 and 10 per cent. of the oil, and in only 2 cases was it over 10, and even then did not exceed 12.

It will be seen from the tabulated results that we have made various other classifications of the linseed cakes examined, both as regards purity and as regards the

country in which they were manufactured. Out of 178 English-made cakes only 36 gave fatty acid exceeding 5 per cent. of the total oil present, and only 10 gave more than 10 per cent.; 43 Russian-made cakes averaged over 10 per cent.; 11 American cakes nearly 9, and 6 Indian-made cakes over 16, only 2 samples being under 10 per cent., while of the others the proportion was high. Generally speaking, it would seem as though a voyage—and especially a tropical voyage—tended to cause some degree of “heating,” and consequent production of acidity.

Out of 268 linseed cakes made from pure—that is, well-screened—linseed, 170 samples had an acidity not exceeding 5 per cent. of the total oil present, while 69 more had over 5, but under 10 per cent. Only 29 samples exceeded 10, and of these only 15 were above 15 per cent. Only 10 exceeded 20 per cent., and 1 only of these was English. On the other hand, 71 cakes made from impure linseed averaged an acidity of over 15 per cent. of the total oil, no fewer than 21 having over 20 per cent. The impurities present were mainly weed-seeds, either incidental to the seed itself and not screened out, or added in the form of screenings from the cleaning of other seed. General mill refuse was also present in some samples, but we regret that our notes do not include detailed memoranda as to the nature of the impurity in individual cases. As a general rule, “dirty,” impure cakes show higher acidity than pure cakes, and when high acidity is found in pure cakes, it is, probably, generally due to bad condition, such as that produced by “heating” or mouldiness. It is not an uncommon thing for mouldy cakes to be “brushed,” and so superficially cleaned from mould, and in these cases a high acidity may arouse suspicion on the part of the analyst, and make him go further into the matter.

As to the general question of the bearing of the presence of much free fatty acid on the value of a food, it would appear to be of small consequence except in so far as it may indicate bad condition due to fermentations which for other reasons may render food unwholesome; for it will be seen that many foods, such as ricemeal,* bran, oatmeal, various kinds of flour, malt-dust, dried grains, and other foods of assured value in every-day use, will be seen to contain a good deal, proportionately speaking, of free fatty acid in their oily matter.

Other points of interest may suggest themselves during the perusal of the tabulated results.

It should be stated that in all cases the “free fatty acid” is, for simplicity’s sake, given in terms of oleic acid, obtained by merely multiplying by 5 the quantity of potassium hydrate required to neutralize the oil.

	Percentage of Moisture.	Per- centage of Oil.	Fatty Acid in 100 parts Oil.	Fatty Acid in 100 parts Cake.
LINSEED CAKES:				
451 various	11·4	10·7	7·5	·79
268 pure linseed	11·4	11·0	5·7	·60
In these 268 pure cakes the range of acidity was as follows:				
170 samples	—	—	5·0 or under	—
69 „	—	—	5 to 10	—

* Rice-oil appears normally to be very rich in free fatty acid, as originally pointed out by Mr. Smetham.

				Percentage of Moisture.	Per- centage of Oil.	Fatty Acid in 100 parts Oil.	Fatty Acid in 100 parts Cake.
14 samples	—	—	10 to 15	—
5 "	—	—	15 to 20	—
7 "	—	—	20 to 30	—
2 "	—	—	30 to 40	—
1 "	—	—	40 to 50	—
71 impure linseed cakes	11.2	10.3	15.4	1.56
In these 71 impure cakes the range of acidity was as follows :							
8 samples	—	—	5.0 or under	—
23 "	—	—	5 to 10	—
15 "	—	—	10 to 15	—
4 "	—	—	15 to 20	—
12 "	—	—	20 to 30	—
6 "	—	—	30 to 40	—
1 "	—	—	40 to 50	—
1 "	—	—	50 to 60	—
1 "	—	—	70 to 80	—
116 freshly-made linseed cakes	—	11.0	3.1	.32
In these 116 samples the acidity ranged as follows :							
103 samples	—	—	5 or under	—
11 "	—	—	5 to 10	—
2* "	—	—	10 to 12	—
<i>Samples arranged according to Nationality of Make :</i>							
178 ENGLISH linseed cakes	—	11.1	4.2	.45
In these 178 samples the acidity ranged as follows :							
142 samples	—	—	5 or under	—
26 "	—	—	5 to 10	—
	—	—	10 to 15	—
	—	—	15 to 20	—
	—	—	20 to 30	—
	—	—	30 to 40	—
120 English cakes made from pure seed	—	11.5	3.9	.44
In these 120 samples the acidity ranged as follows :							
97 samples	—	—	5 or under	—
17 "	—	—	5 to 10	—
5 "	—	—	10 to 15	—
1 "	—	—	20 to 30	—
8 impure English linseed cakes	—	10.1	14.2	1.42
Of these 8 samples, in							
1 sample the acidity was	—	—	under 5	—
3 "	—	—	5 to 10	—
1 "	—	—	10 to 15	—
1 "	—	—	15 to 20	—

* Possibly in these cases the seed itself may have "heated" a little....

	Percentage of Moisture.	Per- centage of Oil.	Fatty Acid in 100 parts Oil.	Fatty Acid in 100 parts Cake.
38 decorticated cakes ...	7.7	11.8	7.5	.84
In these 38 samples the range of acidity was :				
7 samples ...	—	—	5 or under	—
21 " ...	—	—	5 to 10	—
9 " ...	—	—	10 to 15	—
1 " ...	—	—	15 to 20	—
RAPE CAKES :				
46 samples ...	9.4	8.1	7.8	.59
GROUND-NUT CAKES :				
23 samples ...	9.2	9.4	46.0	4.11
In these the acidity ranged thus :				
1 sample ...	—	—	under 10	—
4 " ...	—	—	10 to 20	—
3 " ...	—	—	20 to 30	—
1 " ...	—	—	30 to 40	—
4 " ...	—	—	40 to 50	—
2 " ...	—	—	50 to 60	—
4 " ...	—	—	60 to 70	—
3 " ...	—	—	70 to 80	—
1 " ...	—	—	80 to 90	—
NIGER-SEED CAKES :				
6 freshly-made cakes ...	10.1	6.6	7.4	.50
1 unknown age ...	5.1	16.5	65.1	10.2
5 FRESH PALM-NUT MEALS ...	—	15.1	6.4	.98
1 unknown age ...	—	9.1	52.7	4.80
MAIZE PRODUCTS :				
2 " meals " ...	10.3	6.5	14.5	.93
1 " extract " ...	10.6	8.8	46.2	4.08
1 " bran " ...	9.1	11.4	6.3	.72
3 " germs " ...	10.7	8.4	19.1	1.26
RICE-MEAL :				
13 samples ...	9.4	11.7	69.2	8.38
In these 13 samples of rice-meal the acidity ranged as follows :				
3 samples ...	—	—	30 to 50	—
3 " ...	—	—	50 to 70	—
4 " ...	—	—	70 to 80	—
3 " ...	—	—	over 80	—
3 WHEAT " BRANS " ...	10.7	5.0	53.7	2.62
1 POLLARDS ...	11.4	3.8	40.5	1.80
1 AMERICAN FLOUR ...	12.2	5.0	43.9	2.21
1 FRENCH FLOUR ...	12.5	3.9	46.7	1.87
2 OATMEALS ...	8.3	8.5	36.9	3.15
3 MILLET-MEALS ...	11.2	4.8	35.9	1.61
7 BARLEY-MEALS (fresh) ...	—	2.5	11.6	.25
6 ditto four months old ...	—	2.6	20.4	.44

	Percentage of Moisture.	Per- centage of Oil.	Fatty Acid in 100 parts Oil.	Fatty Acid in 100 parts Cake.
2 BARLEY-MEALS (unknown age)	—	3.9	37.5	1.36
1 LENTIL-MEAL	9.3	1.4	24.4	.33
5 DRIED GRAINS	8.5	8.4	28.3	2.49
3 MALT-DUST	8.5	2.4	32.6	.72
4 COCONUT CAKES	—	16.5	22.0	4.06
1 SESAME CAKE	9.5	13.2	82.5	10.9
1 OLIVE CAKE	10.8	13.4	40.0	5.4
1 SUNFLOWER CAKE	9.5	7.2	6.1	.44
1 COFFEE PARCHMENT	9.9	1.1	19.0	.20
20 samples of feeding meal made from mixtures of linseed and farinaceous meals, etc. :				
When fresh	—	4.1	11.0	—
After six months' keeping	—	4.1	33.2	—
UNCRUSHED SEEDS :				
Linseed (13 samples)	6.3	39.7	1.30	.52
Lowest	—	—	.50	—
Highest	—	—	2.50	—
Cotton-seed (6 samples)	9.8	21.3	7.4	1.7
Lowest	—	—	3.6	—
Highest	—	—	12.6	—
Rape-seed (4 samples)	5.8	39.5	.81	.30
Lowest	—	—	.15	—
Highest	—	—	1.15	—
"Ravison" rape (6 samples)	7.7	25.4	.90	.22
Lowest	—	—	.50	—
Highest	—	—	1.87	—
"Jamba" rape (2 samples)	7.1	25.7	2.4	1.0
Niger-seed (2 samples)	7.3	38.0	.90	.52
Safflower-seed	6.2	29.2	1.9	.55

A large number of "compound" cakes and meals have been tested, but absence of information as to their composition detracts from their interest, and they are here omitted.

On the Analysis of the Nitrogenous Constituents in Meat Extracts and Commercial Peptones. A. Stutzer. (*Zeit. anal. Chem.*, 1895; Drittes Heft, 372-380).—During the last two years much attention has been given to this subject, and results have been obtained which have caused the author to make special investigation with regard to the following points :

1. Has glycogen any influence in the viscosimetrical examination of gelatin?
(See *Zeit. anal. Chem.*, vol. xxxi.)
2. Are gelatin peptones soluble or insoluble in absolute alcohol?
3. Can gelatin be estimated by means of alcohol, as is asserted by Kemmerich and Denaeys? (*Journ. de Pharm. d'Anvers*, January, 1894).
4. Are ammonium salts, when present, soluble or insoluble in alcohol, and how is their separation to be effected?

Glycogen.—At 10° C. the viscosimeter number for water = 52.5. A 2 per cent. solution of glycogen gave 53.5, and a 10 per cent. solution of serum peptone = 60.5. As the amount of glycogen in a meat extract only amounts to from 0.05 to 0.15 per cent., no serious effect on the results need be feared from its presence.

* *Gelatin Peptone.*—The author confirms his previous statement, which had been called in question by Kemmerich and Denaeyer. The small amounts of gelatin peptone in meat extracts are precipitated simultaneously with the gelatin by alcohol, and they may be suitably estimated together.

Estimation of Gelatin.—For the viscosimetrical method considerable quantities of the preparation are required. For the alcohol method of Denaeyer and Kemmerich much smaller quantities suffice, but the analytical process is tedious, and is admitted by Kemmerich to be not sufficiently accurate. The author has found that Beckmann's method (*Anal.*, xx. 44) is not satisfactory, since albumose peptones are precipitated even more readily than gelatin by formaldehyde. Repeated experiments have proved that the alcohol method, when carried out as described below, accounts for 95 to 98 per cent. of the gelatin nitrogen.

Ammonium Salts.—These are completely soluble in alcohol, and apparently partially volatile with alcohol vapour. With regard to amido compounds, the author has made no further investigation, but brings forward the suggestion that compounds of ammonia with fatty acids may be present in meat extract. Ammoniacal nitrogen is estimated by dissolving 10 grammes of the extract in water, and distilling off the ammonia after the addition of barium carbonate. In this case magnesia must not be used.

METHOD OF ANALYSIS.

I. *Estimation of Water, Ash, Sodium Chloride, and Total Nitrogen.*—From 5 to 7 grammes of the dry, and from 20 to 25 grammes of the fluid, preparations are used in these estimations. The amount taken for the water determination is weighed into a thin tinfoil basin (about 20 mm. high and 55 mm. in diameter), dissolved in a little hot water, and ignited fibrous asbestos added in sufficient quantity to almost completely absorb the liquid. The basin is then heated in the water-oven until the weight is constant. The weight of the tinfoil and of the asbestos being deducted, the amount of water is obtained. The tin basin and its contents are subsequently used in the estimation of the gelatin (see IV.).

II. *Nitrogen in the Form of Unaltered Albuminoids, Coagulable Albumin, and Flesh Powder.*—Certain preparations contain small quantities of finely-powdered flesh. This is tested for by treating the meat extract with cold water, and examining the insoluble portion under the microscope. If meat fibre be present, the following method of procedure is adopted: 5 grammes of dry preparations, 8 to 10 grammes of those in the form of extract, or 20 to 25 grammes of fluid preparations, are extracted with cold water, the insoluble matter collected on a filter, and the nitrogen in this determined (meat-fibre nitrogen with very slight quantities of other unaltered albuminoids). The filtrate is acidified with acetic acid, boiled, and filtered. The nitrogen (in the form of coagulable albumin) is determined in the insoluble portion. If no fibrin be present, as in all the better meat extracts and peptones, a weighed

quantity of the extract is treated with water and acetic acid, and the nitrogen determined in the insoluble portion. The filter-paper used must be as free from nitrogen as possible. The author recommends that of Schleicher and Schüll.

The filtrate may also be made up to definite volume, and the nitrogen determined in an aliquot portion. The difference between the result and that of the total nitrogen gives the amount of nitrogen present in the form of albumin.

III. *Nitrogen in the Form of Ammonium Salts.*—Five grammes of the dry substance, 10 grammes of the extract, or 20 to 25 grammes of the fluid preparation, are dissolved in water, and the ammoniacal nitrogen distilled after the addition of barium carbonate.

IV. *Gelatin Nitrogen.*—The tinfoil containing the dry residue left from the water estimation in I., together with the asbestos, is cut into small strips and washed in a beaker four times with absolute alcohol. After removal of the alcohol, the mixture of tinfoil, asbestos, and gelatin is treated with ice-water, to which 10 per cent. of alcohol has been added, the temperature being kept below $+5^{\circ}$ C. by the introduction of small pieces of ice. After being shaken for two minutes in a suitable apparatus, the extraction with ice-water is repeated. The insoluble portion (with the tinfoil and asbestos) is then collected on a filter and washed with ice-water until the filtrate is colourless. The residue, after being boiled up with water in a beaker, is filtered, the filtrate concentrated by evaporation, and the nitrogen it contains estimated by Kjeldahl's method.

V. *Nitrogen in the Form of Flesh Bases and Decomposition Products soluble in Alcohol.*—Five grammes of the dry preparations are warmed in a beaker with 25 c.c. of water. In the case of preparations in the form of extract, the amount taken is about 10 grammes with 10 c.c. of water. With the fluid preparations, of which from 20 to 25 c.c. are taken, no water is required. Thin peptone solutions should be concentrated by evaporation on the water-bath, 50 c.c. being brought to about 25 c.c.

To the solutions 250 c.c. of absolute alcohol are gradually added with constant stirring, this being continued for some minutes after each addition of the alcohol. After 10 or 12 hours the liquid is filtered, and the residue repeatedly washed with alcohol. Leucin, tyrosin, and other decomposition products, together with a part of the flesh bases, will be in solution. The alcohol is completely removed by distillation, the residue dissolved in water, any insoluble matter being filtered off, and the nitrogen it contains estimated, and added to the albumose nitrogen subsequently determined.

The clear solution is diluted with water to 500 c.c., of which 100 c.c. are taken for the total nitrogen present, and a similar quantity for the determination of ammoniacal nitrogen.

The amount of ammoniacal nitrogen found is deducted from the previous result, and gives the nitrogen present in the form of flesh bases and decomposition products.

VI. *Treatment of the Residue insoluble in Alcohol.*—The filter containing the insoluble residue in V. is washed with water into a beaker, the alcohol completely evaporated on the water-bath, and the liquid filtered. A small portion of the albumose usually becomes insoluble by the action of the alcohol, and the nitrogen in this residue must be determined and added to the albumose nitrogen subsequently found.

The filtrate is made up to 500 c.c., of which 50 c.c. are taken for the total

nitrogen present, 50 c.c. for the albumose, gelatin, and peptone, and 100 c.c. for the peptone alone.

The rest of the liquid is concentrated by evaporation to a small bulk, and examined qualitatively for peptone as follows: The albumose and gelatin are precipitated by adding ammonium sulphate, until a little remains undissolved, and filtered off. A few drops of a very dilute solution of copper sulphate are added to the filtrate, followed by a considerable quantity of sodium or potassium hydroxide. Care must be taken to avoid having too much copper, which would prevent the characteristic red coloration from being recognised.

VII. *Pancreas Peptone*.—The solution obtained in VI. contains, besides gelatin and albumose, the entire pancreas peptone. One hundred c.c. of the aqueous solution are concentrated by evaporation to about 8 or 10 c.c. When cool, at least 100 c.c. of a cold saturated ammonium sulphate solution are added, the liquid well stirred, and the precipitate collected on a filter and washed with saturated ammonium sulphate solution. The precipitate is then dissolved in boiling water, and the solution evaporated to dryness with sufficient barium carbonate to expel all the ammonia. The residue is washed with water, the barium sulphate and carbonate filtered off, and the nitrogen found in the filtrate reckoned as being present in the form of pancreas peptone.

VIII. *Albumose Peptone*.—A small quantity will have been found in V. and VI., but the bulk is present in the solution in VI. Fifty c.c. of this solution are mixed with an equal volume of dilute sulphuric acid (1 : 3) in the cold, and phosphotungstic acid added until no further precipitation occurs. The precipitate is washed with dilute sulphuric acid, and the nitrogen in the precipitate determined. This consists of nitrogen in the form of albumose, pancreas peptone, and gelatin, the two former of which have already been determined. The difference gives the nitrogen in the form of albumose, and to this must be added the small quantities found in V. and VI.

IX. *Nitrogen in the Form of Flesh Bases insoluble in Alcohol*.—This is obtained by taking the difference between the total nitrogen of VI. and that found in VIII. after precipitation with phosphotungstic acid.

C. A. M.

On the Fat, Sugar, and Tannin in Coffee Beans. E. Herfeldt and A. Stutzer. (*Zeit. angew. Chem.*, 1895, pp. 469-471.)

Fat.—According to the researches of A. Hilyer (*Forsch. üb. Lebensm.*, 1894, 42), the fat of the coffee-bean consists almost exclusively of olein, with slight traces of free oleic acid. The authors have examined three different kinds of coffee before and after roasting, with the following results:

	SANTOS.		NEW GRANADA.		JAVA.	
	Raw.	Roasted.	Raw.	Roasted.	Raw.	Roasted.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Water	10.86	2.43	10.45	2.18	10.05	2.96
Ash	3.75	4.25	3.40	4.03	3.98	5.33
Organic matter	85.39	93.32	86.15	93.79	85.97	91.71
Containing:						
Fat	8.15	16.58	13.10	15.44	14.00	11.30
Nitrogenous matter	15.93	(2.55) —	12.62	(2.0) —	13.25	(2.12) —
(N × 6.25)						
Non-nitrogenous matter	61.31	—	60.43	—	58.72	—

The ash contained :	SANTOS.		NEW GRANADA.		JAVA.	
	Raw. Per cent.	Roasted. Per cent.	Raw. Per cent.	Roasted. Per cent.	Raw. Per cent.	Roasted. Per cent.
Potash	2.55	—	1.81	—	—	—
Phosphoric acid ...	0.44	—	0.42	—	—	—
Iodine No. of fat ...	88.9	82.4	85.3	86.7	79.7	87.6
Saponification No. ...	183	188	185	217	195	169

The loss on roasting 125 lbs. of the raw beans amounted to about 18 per cent. in each of the three samples.

		SANTOS.			
125 lbs. raw contained	10.18 lbs. fat.	
102 „ roasted „	16.91 „	
		Increase	...	6.73	= 66 per cent.
		NEW GRANADA.			
125 lbs. raw contained	16.37 lbs. fat.	
102 „ roasted „	15.82 „	
		Loss	...	0.55	= 3 per cent.
		JAVA.			
125 lbs. raw contained	17.50 lbs. fat.	
102 „ roasted „	11.52 „	
		Loss	...	5.98	= 33 per cent.

It is difficult to see from what source fat was produced in Santos coffee during roasting, and the authors throw no light on the subject.

Sugar.—The authors' experiments confirm the general belief that ready-formed sugar is not present in the unroasted coffee-bean. The probability is that it only exists as a glucoside in the coffee tannin.

Coffee Tannin.—As the aroma of the roasted beans is said to stand in direct relation to the amount of coffee tannin, several attempts were made to estimate the latter quantitatively. The necessary coffee tannin was prepared by extracting 250 grammes of ground raw coffee with 1 litre of 40 per cent. alcohol for two hours under a reflux condenser, filtering, precipitating the tannin with lead acetate, and again filtering. The lead compound was then thoroughly extracted with alcohol, and the lead removed by sulphuretted hydrogen. Complete desiccation of the filtered tannin being difficult, the alcohol was expelled by heating on the water-bath, and the residue taken up with hot water, the solution being made up to 500 c.c. Twenty-five c.c. were then evaporated in a platinum basin containing ignited sand, and the residue dried and weighed. The mean of several determinations not differing very widely was 0.429 gramme of coffee tannin (including impurities).

In order to obtain the relation between the coffee tannin and the sugar, 25 c.c. of the solution (= 0.429 gramme solid matter) were pipetted into a beaker, and 5 grammes of solid tartaric acid added, followed by 75 c.c. of water. The mixture was warmed for three hours on the water-bath, with occasional renewal of the water, and then neutralized. The solution was added very gradually to 50 c.c. of Fehling's solution and boiled for three minutes, the precipitated cuprous oxide being reduced in hydrogen and weighed as metallic copper. Duplicate results gave 0.0396 gramme and 0.0455 gramme of copper. This want of agreement may possibly be explained by the readily varying conditions of experiment.

Finally, the authors attempted to effect an estimation by decomposing the coffee tannin with bromine-water, and weighing the bromo-caffeic acid in a Gooch's crucible, but concordant results were not obtainable.

C. A. M.

Water in Honey. J. Graftian. (*Bull. de l'Assoc. belge des Chim.*, 1895, iv., pp. 118-120.)—The amount of water in honey varies very considerably in natural specimens according to the dryness of the season in which it was produced and the conditions of storage. The degree of completion of the honey-comb has also considerable influence, there being naturally much less evaporation where most of the cells are sealed. In eight samples of honey examined by the author, the following percentages of water were found: 17.70, 21.75, 14.89, 13.89, 14.53, 16.93, 22.23, 19.50. In estimating the water, about 5 grammes are weighed on a spiral of filter-paper 20 to 25 mm. wide and 30 cm. long, which has been previously dried and weighed. Ten c.c. of hot water are added to distribute the honey, and the spiral dried in a flat-bottom basin until the weight is constant.

C. A. M.

The Estimation of Glycerin in Wine and Beer. A. Partheil. (*Arch. Pharm.*, 1895, ccxxxiii., 391; through *Chem. Zeit. Rep.*, 1895, 291.)—Fifty c.c. of the liquid to be examined, after addition of a pinch of calcium carbonate, are evaporated down to 10 or 15 c.c., and introduced into a stoppered 100 c.c. retort. In place of the stopper a soft cork is used, through which passes a vaselined glass rod. The retort is erected inside an air-bath, the bottom of which consists of a plate of sheet-iron, the sides and top being made of asbestos card. The neck is connected with a globular receiver, placed in a large funnel, the second opening of the receiver being joined to an inverted condenser, and then to a pump. The cooling water of the condenser is arranged to run over the receiver. In the air-bath is fixed a thermometer. The liquid is distilled, under ordinary pressure, almost to dryness, till the temperature reaches 120° C. After cooling to about 60° C., the pump is set in action, and the distillation continued for one and a half hours at 180° C. at a pressure of 25 to 30 mm. The pressure is then released, the retort cooled, 10 c.c. of water added, and the liquid again distilled at 120°, without the pump, as far as possible. The distillate is diluted to about 200 c.c., 8 to 10 grammes of solid caustic soda dissolved in it, and 5 per cent. permanganate added till the colour remains bluish-black. It is heated for an hour on the water-bath, decolorized with sulphurous acid, 20 c.c. of glacial acetic acid added, heated till all SO₂ is driven off, again diluted to about 200 c.c., and the oxalic acid thrown down with calcium chloride. The precipitate is collected on an asbestos filter, washed thoroughly, dissolved in dilute sulphuric acid on the water-bath, and the oxalic acid in it determined by titration with standard permanganate. The process is stated to be very exact.

F. H. L.

The Estimation of Zinc in Desiccated Apples. L. Legler. (*Chem. Zeit.*, 1895, xix., 1763.)—Fifty grammes of the sample are well dried in a porcelain basin, and carbonized by the application of a gas-flame from above. The carbonaceous residue is broken up, digested in moderately strong hydrochloric acid, the liquid diluted, filtered, made alkaline with excess of ammonia, diluted again to a definite

volume, and the zinc estimated in a portion by precipitation with sulphuretted hydrogen in the presence of acetic acid. The figures given by this process agree well with those obtained by treating the apples with oxidizing materials, and apparently there is no loss of zinc by volatilization.

F. H. L.

The Acetyl Numbers of Lard, Cotton-seed Oil, and Tallow. E. Spaeth. (*Forschungber. Lebensm. Hyg.*, etc., 1895, ii., 226; through *Chem. Zeit. Rep.*, 1895, 292.)—The author quotes a number of figures which show that the determination of the acetyl number is practically useless for the detection of cotton-seed oil in lard.

F. H. L.

On the Chemical Recognition of Horse-flesh. W. Niebel. (*Zeit. Fleisch u. Milchhyg.*, 1895, 86; through *Chem. Centr.*, 1893, 323).—The author criticizes the method of Edelmann and Brautigam, on the ground that the reaction with iodine is uncertain, since glycogen is also found in the flesh of dogs, cats, and very young calves, in the livers of cattle, and in meat-extract to the amount of 1.5 per cent. In old sausages from horse-flesh these chemists always obtained the glycogen reaction, although that substance would usually be completely decomposed under these circumstances. There is also an uncertainty in the reaction caused by the fact that the dextrins resulting from the starches give a similar coloration with iodine, and no means of removing them is known. The red colour with iodine is not sufficient proof of the presence of glycogen, which should be isolated in pure condition. Nevertheless, the iodine coloration, and the occurrence of more than 1 per cent. of grape sugar in the fat-free substance, points to the presence of horse-flesh in a sample, even when all the glycogen has been decomposed. The red colour only fails in the case of young foals.

C. A. M.

The Detection of Oil and Gurgun Balsam in Copaiba. E. Hirschsohn. (*Pharm. Zeits. Russ.*, 1895, xxxiv., 497 and 499; through *Chem. Zeit. Rep.*, 1895, 274.)—To detect fatty oils, twenty to forty drops of the balsam are boiled for some time with 1 to 2 c.c. of a solution of one part of caustic soda in five parts of 95 per cent. alcohol. A turbidity is produced in the presence of 10 per cent. of oil, while 20 or 30 per cent. is shown by the liquid becoming gelatinous. The mixture must also bear the addition of two volumes of ether, and even cooling to 0° C., without any precipitate of a gelatinous character appearing. When one volume of the copaiba is added to three of 90 per cent. alcohol, a liquid is formed from which no drops of oil must separate after one hour's standing.

Gurgun balsam is found by its characteristic colour reaction with stannous chloride. One volume of the suspected balsam is heated with three volumes of 95 per cent. alcohol and 1 gramme of crystallized stannous chloride till dissolved. One per cent. of Gurgun causes a rose colour, becoming violet after a time; with 10 per cent. a bright red is produced at once, changing in half an hour to violet and then blue.

F. H. L.

Kjeldahl's Method for the Determination of Nitrogen. Bernard Dyer, (*Journ. Chem. Soc.*, vols. lxvii., lxviii., pp. 811-817.)—The Kjeldahl method, being free from the errors attendant on the soda-lime process of nitrogen determination, is, in

its modified forms, superseding the latter in the examination of feeding-stuffs and fertilizers, being also capable of application in cases where nitrates are associated with ammonia. In its original form the method consisted in oxidizing the substance under examination by means of strong sulphuric acid at boiling temperature, and employing potassium permanganate to complete the action. This, however, gave low results and was somewhat slow, although its course could be accelerated and the results brought out better by the addition of a small quantity of mercury or mercuric oxide. The introduction by Gunning of potassium sulphate into the sulphuric acid during the process of oxidation facilitated the action of the acid by raising the boiling-point, and thereby reducing the time required for completing the oxidation, the addition of mercury affording still further assistance in this latter direction.

This modification, the "Kjeldahl-Gunning-Arnold" process, is suitable for feeding-stuffs or fertilizers containing no nitrates. It is performed by treating from 0.5 to 5 grammes of substance with about 20 c.c. of strong sulphuric acid—with the addition of 1 drop of mercury—in a Jena bulb flask fitted with a loose balloon-stopper. Heat is applied gently until vigorous action has ceased, whereupon the temperature may be slowly raised to bring the liquid to a brisk boil. In about fifteen minutes 10 grammes of potassium sulphate are introduced, and the whole is then boiled until clear and without colour, an operation lasting from half an hour to an hour, according to the quantity of carbonaceous matter present. As the sulphuric acid condenses on the pendant of the bulb-stopper, and returns to the liquid, but little is lost, except in the form of sulphurous acid. The contents of the flask are then transferred to a large Jena distilling-flask, connected with a condenser, and also fitted with a tapped funnel for the admission of an excess of sodium hydrate solution, together with a little sodium sulphide for decomposing any compounds of nitrogen and mercury where this metal has been employed. Some zinc-foil having been inserted to prevent bumping, the liquid is distilled, and the condensed vapours collected in a measured quantity of standard acid, the ammonia being determined by titration. In order to prevent soda-spray passing over with the steam, an arched tube of block-tin, rising to a vertical height of some 15 to 18 inches from the cork of the still, is recommended for connecting the latter with the receiving-flask (which should stand in a tank of running water) by way of a pear-shaped adapter, provided with a large expansion, to allow for alterations of pressure, and prevent the acid being drawn back over into the distilling-flask.

The margin of error due to nitrogenous impurities in the reagents, and to the action of steam on the glass, seldom exceeds 0.2 c.c. to 0.4 c.c. of $\frac{1}{10}$ normal sodium hydrate solution (= 0.0003 to 0.0005 gramme of nitrogen), and should be determined by a blank experiment whenever fresh supplies are taken in.

In the case of substances containing nitrates as well as organic or ammoniacal nitrogen, the total nitrogen may be accurately ascertained by Jodlbauer's modification (the "Kjeldahl-Gunning-Arnold-Jodlbauer" process), wherein about 2 grammes of phenol—or, better, salicylic acid—are added to the sulphuric acid before commencing, between 1 and 2 grammes of zinc-dust and 1 drop of mercury being dissolved in the contents of the flask previous to applying heat. By proceeding in the same manner as before, the whole of the nitrogen is then converted into ammonia. The author has

found it necessary to pour the oxidizing agents on to the substance *quickly* in order to prevent the loss from the formation of lower oxides of nitrogen, and the consequent depreciation of the results, occurring when the addition is made from a pipette in the usual way. This precaution enables the whole of the nitrogen, even from ammonium nitrate, to be retained in solution, and its adoption will probably lead to the disappearance of the discrepancies frequently observed in the results of analyses of compound fertilizers and mixtures of salts of ammonia and nitrates of the alkalies.

Tables are given showing the degree of accuracy attainable by the modified methods above described, in more than 40 different pure nitrogenous organic compounds, alkali nitrates, etc. The method fails, however, to give satisfactory results with phenyl-hydrazine salts, although hydrazobenzene gives the theoretical yield of nitrogen. For hydrazine salts Dafert suggests a preliminary reduction by sugar in presence of sodium acetate before adding sulphuric acid. C. S.

The Analysis of Glue and Glue-yielding Substances. W. Fahrion. (*Zeit. angew. Chem.*, 1895, pp. 529-530.)—The substance to be examined is finely rasped, and two portions of from 3 to 5 grammes each weighed out. In one the moisture is determined by drying at 110° to 120° C. until the weight is constant. The residue is then used for the determination of ash.

The second portion is mixed with 15 to 25 c.c. of an 8 per cent. solution of alcoholic soda, and evaporated to dryness on the water-bath. The residue is taken up with alcohol, and again brought to dryness. It is then washed with hot water into a separating-funnel, acidified with hydrochloric acid, and on cooling shaken out with ether. The solid oxy-acids which are left undissolved may be estimated by dissolving them in warm alcohol, evaporating the latter, and weighing the residue. On evaporating the ethereal extract, the unsaponifiable matter, the fatty acids, and the fluid oxy-acids are obtained. The residue is weighed, and then treated with petroleum spirit, in which the fluid oxy-acids are insoluble. On shaking the petroleum spirit solution in a separating-funnel with a soda solution containing some alcohol, the fatty acids are removed, while the unsaponifiable matter remains in solution, and may be weighed after evaporating the petroleum spirit. The alkaline solution containing the fatty acids is heated on the water-bath to remove alcohol, the residue diluted with water, decomposed in a separating-funnel with hydrochloric acid, and shaken out with petroleum spirit, which on evaporation leaves the fatty acids. The following substances were examined by this method: (1) A very fine white glue; (2) Hide-powder; (3) Purified sheep-leather; (4) Sheep's horn; (5) Bone belonging to the horn.

The results obtained were:

	Water.	Ash.	Unsaponifiable Matter.	Fatty Acids.	Fluid Oxy-acids.	Solid Oxy-acids.	Proteid Substance.
Glue ...	13.74	1.80	0.49	0.08	0.04	0.27	83.58
Hide-powder ...	19.15	0.25	0.72	0.18	0.08	0.87	79.25
Purified leather ...	11.23	10.06	9.74	0.99	0.46	1.01	66.51
Sheep's horn ...	9.09	1.00	0.68	1.03	0.29	1.49	87.62
Bone ...	10.00	58.87	4.81	4.23	0.19	1.52	25.38

The proteid substance was simply determined by difference. With regard to the nature of the oxy-acids and the unsaponifiable matter, a further communication is promised. The behaviour of the constituents towards various solvents is noteworthy. The proteid acid resulting from the action of the alcoholic potash on the original substance is soluble in water, alcohol, and alkalies, insoluble in ether and petroleum spirit. The solid oxy-acids are soluble in alcohol and alkalies; insoluble in water, ether, and petroleum spirit. The fluid oxy-acids are soluble in alcohol, ether, and alkalies; insoluble in water and petroleum spirit. The fatty acids are soluble in alcohol, ether, petroleum spirit, and alkalies; insoluble in water. The unsaponifiable matter is soluble in ether and petroleum spirit, partially soluble in alcohol, but insoluble in water and alkalies.

C. A. M.

A Method for the Quantitative Estimation of Ammonia in Tobacco in the Presence of Nicotine. Viktor Vedrödi. (*Zeit. anal. Chem.*, 1895; Viertes Heft, pp. 413-420.)—In a previous communication, the author stated that in Kissling's (*Zeit. anal. Chem.*, xxxii. 567) method of estimating nicotine too high results were obtained, this being probably due either to the decomposition of part of the albumin in the tobacco into ammonia, or to part of the ammonia passing into the ethereal extract. To this criticism Kissling replied that the fault lay in the manner of working, and not in any inherent defect in his process. To settle the question, the author has made further experiments, using tobacco-powder which had been deprived of its ammonia and nicotine, and to which a known quantity of nicotine had subsequently been added. The results obtained were too high, this being the more so the longer the distillation in the current of steam was continued. The explanation now suggested is that small particles of the sodium hydrate are carried over mechanically.

In order to avoid this source of error, the following modification of Kissling's method is proposed: The ethereal extract is mixed with about 100 c.c. of water, and first the ether, and subsequently the water, distilled without the application of a current of steam except at the very end to remove the last traces of nicotine. The distillate is titrated with $\frac{1}{2}$ normal sulphuric acid, 1 c.c. of which corresponds to 0.0566 gramme nicotine.

In 20 grammes of tobacco-powder, freed from nicotine and ammonia, and mixed with 10 c.c. of a nicotine solution (= 0.3 gramme nicotine), the amount of the alkaloid found in ten experiments varied from 0.282 to 0.3 gramme, this being at the most a loss of 0.09 per cent. The distilled ether should also be titrated, since a small amount of nicotine passes over with it.

In the author's opinion, the amount of ammonia has a great influence on the pungency of tobacco, and it is therefore essential to estimate this constituent as well as the nicotine. Kissling estimates it by treatment of the ethereal solution with platinic chloride, but this is only calculated to give the amount of ammonia formed by the decomposition of the nicotine in the solution.

The author finds that, after determining the nicotine, the ammonia may be accurately estimated by mixing 20 grammes of tobacco-powder with 10 c.c. of alcoholic soda and 100 c.c. of water, distilling to dryness, and driving over the last traces by

means of a current of steam. The distillate is titrated with standard sulphuric acid, and from the result, after deducting the amount of acid required by the nicotine in the previous estimation, the quantity of ammonia may be calculated. The flask found most suitable for the distillations had a capacity of from 600 to 700 c.c., with a neck 9 cm. long and 4 cm. in diameter. Control experiments gave results as satisfactory as in the case of the nicotine estimations.

C. A. M.

Experiments on Tannin. L. Fajans. (*Zeit. angew. Chem.*, 1895, pp. 471-472.)

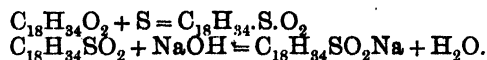
—Attempts made by the author to estimate tannin quantitatively by oxidation with nitric acid, though not yet very successful, seem to point to the conclusion that such a method is possible if once the conditions of oxidation can be determined. In the experiments nitric acid of varying strengths was used, the tannin oxidized directly and in acetic acid solution, and the oxalic acid determined sometimes gravimetrically, and sometimes by titration with permanganate. The results obtained are tabulated as follows :

In the first series of experiments in which nitric acid 25° Beaumé at 17·5° C. was employed, the oxalic acid, as determined by titration, amounted to from 46·98 to 52·9 per cent. In the second set nitric acid 33° Beaumé was used, and oxalic acid amounting to from 50·56 to 55·72 per cent. was found by a gravimetric determination. In a third series, a slightly stronger nitric acid was employed, 35° B., and amounts varying from 52·78 to 60·94 per cent. of oxalic acid were obtained by titration. In series four, acid of the same strength was employed, the oxalic acid being determined gravimetrically. The amounts found varied from 47·71 to 57·51 per cent. It was found that fuming nitric acid could not be used, as it attacked the oxalic acid formed. Some interesting results were also obtained by the action of other oxidizing agents : Tannin heated with potassium chlorate solution on the water-bath, and the liquid subsequently treated with hydrochloric acid, yielded oxalic acid. On the other hand, gallic acid, which, when oxidized with nitric acid, yields oxalic acid, when treated with potassium chlorate and hydrochloric acid, gave not oxalic acid, but isotrichlorglyceric acid ($C_3H_3Cl_3O_4$). Oxalic acid was formed as a by-product when an acid solution of tannin was decomposed by potassium permanganate. The same substance was also produced in alkaline solution by warming the solution of tannin with potassium ferricyanide and adding caustic potash to alkaline reaction. On decomposing the diluted liquid with acetic acid, a considerable quantity of oxalic acid was found in the filtrate. Sodium peroxide likewise produced oxalic acid, but the author gives a warning with regard to its use, a violent explosion having occurred on warming 0·3 grammes of tannin with the same quantity of peroxide and a drop of water in a long-necked flask.

C. A. M.

The Action of Sulphur on Unsaturated Fatty Bodies. Julius Altschul. (*Zeit. angew. Chem.*, 1895, pp. 535-542.)—In the combination which takes place between sulphur and unsaturated fatty bodies, the author shows that only addition compounds are formed, and that the reaction, which takes place at comparatively low temperatures, is analogous to that occurring with the halogens and oxygen

The final proof of chemical combination is furnished by the saponification of the addition compounds. On this point there have been but few communications, and some uncertainty has been felt on account of statements in German patent literature (*e.g.*, October 12, 1892, R. 7,303, iv. 23). The author proves that a certain quantity of oleic acid, after heating with about 1 per cent. of its weight of sulphur at about 140° C., requires the same amount of alkali for saponification as it did before this treatment :



That the sulphur soap produced is not a substitution compound may be demonstrated by liberating the fatty acids and heating them to about 200° C. A large volume of sulphuretted hydrogen is immediately liberated, which, had substitution previously taken place, would not have been the case. Some details of experiments of the action of sulphur chloride on the compounds of sulphur and unsaturated fatty bodies are also given. The oils chosen were those which readily absorb oxygen—linseed, poppy, cotton, and cod-liver oils. In each case sulphur chloride combined with the sulphur compounds, but the amount required to form a solid substance was considerably less than that required by the untreated oils. Linseed-oil, for example, which untreated was saturated with 25 to 30 per cent. of sulphur chloride, after the addition of sulphur required only 10 to 12 per cent.

C. A. M.

Critical Temperatures of Dissolution : a New Constant for the Examination of Fatty Bodies. L. Crismer. (*Bull. de l'Assoc. belge des Chim.*, 1895, ix. 5, 143-172.)—In a previous communication (see ANALYST, xx. 209) the author gave a short account of this method of examining fats and hydrocarbons. He now goes very fully into the subject, illustrating his paper with curves of the critical temperatures of dissolution. In his opinion, this is completely analogous to the *critical temperature* of the liquefaction of gases. His method of manipulation was given in the former paper.

The results obtained with ten samples of butter were as follows :

Manner of Fusion.	Density at 100° C.	Refractive Index (d'Amagat).	Fatty Acids Insoluble.	Volatile Acids (Reichert-Meissl).	Critical Temperature.	Oil in Margarine.
1. Clear	0.866	-33°	87.56	—	100°	—
2. Turbid	0.864	-23°	90.19	20.6	106.5°	Sesame
3. Clear	0.867	-33°	85.60	—	90°	—
4. „	0.866	-33°	87.24	—	96°	—
5. „	0.8655	-30°	88.33	27.9	99°	—
6. „	0.866	-33°	86.82	—	96.5°	—
7. „	0.866	-32°	87.44	—	99°	—
8. „	0.8665	-34°	87.46	—	96°	—
6. „	0.866	-34°	86.25	—	95°	—
10. „	0.866	-30°	87.96	—	97.2°	—

Some of these results are anomalous. Sample 3 had a very low critical temperature, but the Hehner number was also excessively low. The two temperatures 96.5° and 95° similarly corresponded to equally low amounts of fixed fatty acids.

It thus appears that the critical temperature and the amount of insoluble acids stand in some sort of relationship, and the author's further experiments on other fats and oils completely confirm this.

The critical temperature of dissolution of mixtures is approximately the arithmetical mean of those of its constituents. It may be calculated from the formula—

$$T_m = \frac{nTa + (100 - n)Tb}{100},$$

where

T_m = the critical temperature of the mixture,

T_a = the critical temperature of constituent a ,

T_b = the critical temperature of constituent b ,

n = the volume of constituent a in 100 volumes,

$100 - n$ = the volume of constituent b in 100 volumes.

Thus :

Butter. $T_a = 100^\circ \text{C.}$	Margarine. $T_b = 124^\circ \text{C.}$	T_m calculated.	T_m found.		
1 volume	1 volume	112°	112.8	112.8	112.8
2 "	1 "	108°	109	109	108.5
3 "	1 "	106°	107.5	107	107.5
4 "	1 "	104.8°	105	105	105.2

The alcohol usually employed had a density of 0.8195 at 15.5°C. By using a more dilute alcohol the critical temperature was correspondingly raised. C. A. M.

A Method of Converting the Cuprous Oxide obtained in the Gravimetric Estimation of Sugar into Cupric Oxide. K. Farnsteiner. (*Forschungsber. Lebensm. Hyg.*, etc., 1895, ii., 235; through *Chem. Zeit. Rep.*, 1895, 292.)—When in a sugar determination the cuprous oxide is collected in a Soxhlet filtration tube, the author suggests that it is more expeditious, instead of reducing to the metallic state by means of a current of hydrogen, to oxidize to cupric oxide and weigh as such. After washing the precipitate with alcohol and ether, the end of the tube is connected with the exhaust pump, and a current of air drawn through it. A gas-flame is then cautiously applied, and the cuprous oxide gradually heated to incandescence; when the tube itself and the asbestos have become red-hot, the flame is withdrawn, and the whole allowed to cool. If the tube be made of combustion tubing, there is no fear of fracture. F. H. L.

The Rotatory Power of Maltose. H. Ost. (*Chem. Zeit.*, 1895, xix., 1727.)—The author has redetermined this constant, working on seven different specimens of maltose, in solutions of from 2 to 21 per cent. strength, and at a temperature of 20°C. The value obtained was $+136.95^\circ$. By rejecting two of the preparations on the ground of possible impurity, this becomes $[\alpha]_D = +137.04^\circ$ at 20°C. , with a possible error of $\pm 0.19^\circ$. Meissl (*J. prakt. Chem.*, 1882, xxv., 114) found it to be 138.1 to 138.4; while Tollens and Parcus (*Annalen*, 1890, ccvii., 160) obtained figures agreeing closely with the present result.

The specific gravities at $\frac{20^{\circ}}{4}$ of a number of maltose solutions are given in the following table, the maltose being returned as anhydrous :

Specific Gravity.	Grammes of Maltose per		Specific Gravity.	Grammes of Maltose per	
	100 c.c.	100 Grammes.		100 c.c.	100 Grammes.
1.005	1.78	1.77	1.045	12.10	11.54
1.010	3.07	3.05	1.050	13.89	12.72
1.015	4.36	4.31	1.055	14.68	13.89
1.020	5.65	5.54	1.060	15.97	15.05
1.025	6.94	6.75	1.065	17.26	16.20
1.030	8.23	7.95	1.070	18.55	17.34
1.035	9.52	9.15	1.075	19.84	18.46
1.040	10.81	10.35	1.080	21.13	19.57

F. H. L.

The Estimation of Sugar by Means of Potassium-Copper Carbonate. H. Ost. (*Chem Zeit.*, 1895, xix., 1784 and 1829.)—Since the original publication of this process, it has been reinvestigated, and the exact value of the copper solution as against different varieties of sugar redetermined. In its present form it possesses several advantages over Fehling's process: the carbonate solution has far less action on cane-sugar, the metal reduced per unit of sugar is greater ($1\frac{1}{2}$ to 2 times), the reducing power of the different sugars varies more, and alterations in the time of boiling influence the results less than is the case with the older method. As now prepared, the "strong" copper solution will keep for a year or so if stored in large bottles, without altering in strength, while the "weak" solution may be preserved indefinitely. The former is made up by adding 17.5 grammes of crystallized copper sulphate to a solution containing 250 grammes of normal carbonate, and 100 grammes of acid carbonate of potassium, diluting the whole to 1 litre. Both carbonates must be "chemically pure," especially as regards freedom from silica, and the solution of the copper sulphate should be added gradually to the potassium salts to avoid loss of carbon dioxide. If the liquid is not perfectly clear, it must be filtered either through asbestos or paper, the first portions running through being rejected. The "weak" copper solution is prepared in a precisely similar manner, but contains only 3.6 grammes of copper sulphate per litre. It is very useful for the determination of invert sugar in substances where non-reducing sugars are present, as in beet sugar for instance. The analysis must be so arranged that not more than 30 to 88 milligrammes of the invert sugar are contained in the liquid tested. The weak solution is also an excellent substance for the qualitative detection of reducing sugars.

The process is as follows: 100 c.c. of the (strong) copper solution are rapidly heated with 50 c.c. of the sugar solution in a narrow-necked beaker, and boiled for ten minutes, then cooled as quickly as possible, and filtered through an asbestos tube. If the filtrate is very blue, the precipitate is washed once with a little potassium bicarbonate solution, then with water and alcohol, and finally reduced in a current of

hydrogen free from arsenic. Occasionally it happens that the asbestos is discoloured by a trace of carbon: should there appear to be a ponderable amount, it may be removed by drying the cuprous hydrate very carefully, and then igniting it for a few moments in air (a current is quite unnecessary) before the hydrogen is employed to reduce it to the metallic state. In working with the weak solution, the process is identical, but the boiling is only continued for five minutes. When the sugars examined contain lime, this must be first precipitated by ammonium oxalate, and in cases where lead acetate has been employed, the oxalate can be added at the same time as the sodium sulphate used to remove the excess of lead. Attention must be paid to the quality of the asbestos used in the filter, as some varieties are very readily attacked by alkalis.

The figures in Table III. refer to results obtained with the weak copper solution, Tables I. and II. to the strong. When approximate results only are required, and also when it is desired to work volumetrically, the author prefers still to use Fehling's solution.

The values for maltose in Table II. are given as for anhydrous maltose, while Table III. shows the influence of gradually increasing amounts of cane-sugar on invert sugar.

F. H. L.

TABLE I.

Dextrose and Lævulose.

Copper.	Dextrose.	Lævulose.	Copper.	Dextrose.	Lævulose.	Copper.	Dextrose.	Lævulose.
435	152.3	145.9	315	99.2	92.8	195	59.1	55.6
430	149.8	143.4	310	97.4	91.0	190	57.6	54.1
425	147.3	140.9	305	95.6	89.2	185	56.0	52.7
420	144.8	138.4	300	93.8	87.5	180	54.5	51.2
415	142.3	135.9	295	92.0	85.8	175	53.0	49.8
410	139.8	133.5	290	90.2	84.1	170	51.5	48.4
405	137.3	131.1	285	88.4	82.4	165	50.0	46.9
400	134.9	128.7	280	86.7	80.8	160	48.5	45.5
395	132.5	126.4	275	85.0	79.2	155	47.0	44.1
390	130.1	124.1	270	83.3	77.6	150	45.5	42.7
385	127.8	121.8	265	81.5	76.1	145	44.0	41.3
380	125.5	119.5	260	79.8	74.6	140	42.5	39.9
375	123.3	117.2	255	78.1	73.1	135	41.0	38.5
370	121.1	115.0	250	76.5	71.6	130	39.6	37.1
365	119.0	112.8	245	74.9	70.1	125	38.1	35.7
360	116.9	110.6	240	73.3	68.6	120	36.7	34.3
355	114.8	108.5	235	71.7	67.2	115	35.2	32.9
350	112.8	106.4	230	70.1	65.7	110	33.7	31.6
345	110.8	104.3	225	68.5	64.3	105	32.2	30.3
340	108.8	102.3	220	66.9	62.8	100	30.7	29.0
335	106.8	100.3	215	65.3	61.4	195	29.2	27.7
330	104.9	98.4	210	63.8	59.9	190	27.8	26.4
325	103.0	96.5	205	62.2	58.5	185	26.3	25.1
320	101.1	94.6	200	60.7	57.0	180	24.8	23.8

TABLE II.
Invert Sugar and Maltose (Anhydrous).

Copper.	Invert Sugar.	Maltose.	Copper.	Invert Sugar.	Maltose.	Copper.	Invert Sugar.	Maltose.
435	147·5	263·7	310	94·4	176·3	185	54·5	104·7
430	145·3	259·3	305	92·6	173·3	180	53·1	101·9
425	143·1	255·0	300	90·9	170·3	175	51·6	99·2
420	140·8	250·9	295	89·2	167·3	170	50·2	96·4
415	138·5	247·0	290	87·5	164·4	165	48·7	93·7
410	136·2	243·2	285	85·8	161·4	160	47·3	90·9
405	133·9	239·4	280	84·1	158·5	155	45·8	88·2
400	131·6	235·6	275	82·4	155·5	150	44·4	85·4
395	129·3	231·9	270	80·7	152·6	145	42·9	82·6
390	127·0	228·2	265	79·1	149·7	140	41·5	79·9
385	124·8	224·6	260	77·5	146·8	135	40·1	77·1
380	122·6	221·1	255	75·9	143·9	130	38·6	74·4
375	120·4	217·7	250	74·3	141·1	125	37·2	71·6
370	118·2	214·4	245	72·7	138·2	120	35·8	68·9
365	116·0	211·1	240	71·1	135·4	115	34·3	66·1
360	113·9	207·9	235	69·5	132·5	110	32·9	63·4
355	111·8	204·7	230	68·0	129·7	105	31·4	60·6
350	109·8	201·5	225	66·5	126·8	100	30·0	57·9
345	107·8	198·3	220	65·0	124·0	195	28·5	55·1
340	105·8	195·2	215	63·5	121·2	190	27·1	52·3
335	103·8	192·0	210	62·0	118·4	185	25·6	49·6
330	101·8	188·8	205	60·5	115·7	180	24·2	46·8
325	99·9	185·7	200	59·0	112·9	175	—	44·1
320	98·0	182·5	195	57·5	110·2			
315	96·2	179·4	190	56·0	107·4			

TABLE III.
Mixtures of Invert and Cane Sugars.

[illegible]

The Influence of the Metals of the Platinum Group on the Estimation of Gold by "Parting." E. Priwoznki. (*Oesterr. Zschr. Berg. u. Hüttenw.*, special report; through *Chem. Zeit. Rep.*, 1895, 214.)—In quantities of not more than 2 per cent. the presence of platinum does not vitiate the results obtained in the nitric acid methods for the determination of gold; above that amount the yield of gold is too high. Palladium, being soluble, can have little influence; but iridium, rhodium, and ruthenium are likely to prove more objectionable. The compounds of osmium, being volatile, are removed during the heating. The solubility of platinum in nitric acid in presence of silver is due to the formation of a double salt. F. H. L.

REVIEWS.

AIDS TO THE ANALYSIS OF FOOD AND DRUGS. By T. H. PEARMAIN and C. G. MOOR, M.A., F.C.S. (London: Baillière, Tindall and Cox. 160 pages.) Price 3s. 6d.

This little book is one of the "Students' Aid" Series, and, if we are not mistaken, is based upon a previous work in the same series by a different author.

Messrs. Pearmain and Moor, in their preface, state that no work of moderate size devoted to the analysis of food and drugs has recently been published. They add that the work is not intended to be used as a cram-book for examinational purposes, and we cordially congratulate the authors on their belief, and on their courage in expressing the opinion, that they "cannot emphasize too strongly the fact that food analysis is not to be taught in a few weeks, as is frequently attempted in the interests of public health students."

In their preface the authors acknowledge their indebtedness to the writers of various works on the subject of food and drugs, but when conveying processes from these works to the body of their book, they frequently omit to mention their source.

Both Mr. Pearmain and Mr. Moor are favourably known for their careful work on various kinds of food and drugs, and their extensive laboratory experience in these subjects is an ample guarantee that the processes they describe are in general trustworthy and of a practical kind. Their information is in most respects well up to date, and many original figures are given.

The authors do not limit themselves to articles liable to be taken under the Sale of Food and Drugs Act, but include in their book directions for the analysis of commercial disinfectants, soap, cinchona-bark, urine, etc. Practical methods are given for the examination of baking powders, lime, and lemon-juice, tincture of rhubarb, aromatic spirit of ammonia, and other preparations, which the public analyst is liable to be called on to examine occasionally.

The first section is devoted to milk, which is followed by a welcome section on the analysis of condensed milk. For the determination of the fat in condensed milk, the authors give the preference to the Adams process, having found the Werner-Schmidt method unsuitable, and the Leffmann-Beam results generally below the truth. (This experience, of course, only applies to condensed milk.) Sections dealing with butter, cheese, bread and flour, follow, after which groceries are considered. The section on infants' foods contains valuable information and analytical data not pre-

viously published. The Kjeldahl-Gunning process for the determination of nitrogen is well described.

So far as we have checked their calculations, the authors' figures are generally correct. On page 92 the iodine absorption by Hübl's process should be 74.34, instead of 75.6, as stated. Some other mistakes have crept in, which affect the reliability of certain of the processes. Thus, in the description of the estimation of phosphates by the molybdic acid process on page 62, after obtaining opalescence by addition of aminonia, a direction to add a certain quantity of nitric acid is apparently omitted. On page 53 the authors state that if an alcoholic extract be made of mustard containing turmeric, it will acquire an orange-red colour on treatment with hydrochloric acid, changed to green and blue by excess of caustic soda. It is evident that this refers to the boric acid reaction with turmeric, but no mention of the addition of boric acid or borax is made in the text. On page 127, the authors state that a yield of 7 to 5 volumes of nitrous oxide from spirit of nitrous ether is equivalent to 3 and 2 per cent. respectively of ethyl nitrite, whereas the ratio of 7 to 5 is not the same as that of 3 to 2. However, these and other minor defects do not seriously affect the value of a work which has been carefully compiled, and contains a great deal of useful information conveyed in a concise and practical form. In some cases greater clearness would be desirable, and the authors' English is not infrequently very slipshod.

The work concludes with alcohol tables, weights and measures, co-efficients required in volumetric analysis, tables of atomic weights, and numerous other useful data.

On the whole, Messrs. Pearmain and Moor are to be congratulated on the way in which they have executed their task. In the next edition, which we hope will soon be called for, the authors will doubtless rectify the errors and omissions almost inseparable from the first production of a new work.

A. H. A.

CHEMISTRY OF URINE: A PRACTICAL GUIDE TO THE ANALYTICAL EXAMINATION OF DIABETIC, ALBUMINOUS, AND GOUTY URINE. By ALFRED H. ALLEN. (J. & A. Churchill, 1895.) Price 7s. 6d.

This is an octavo volume of 200 pages, uniform with those other well-known volumes by the same author on "Commercial Organic Analysis." Indeed, this book forms a portion of the concluding volume of that work, and, according to a statement in the preface, is published in a separate form for the benefit of the medical profession, and more especially physicians who may be called upon to advise as to the acceptance or rejection of candidates for life assurance. The author, with this purpose before him, restricts the scope of the work mainly to the pathological conditions of urine attendant upon diabetes, albuminuria, and gout.

The treatment of these three subjects is preceded by two short chapters—one upon the general composition of urine, another upon its preliminary examination, and is followed by a chapter on the colouring matter of urine, the book being brought to a close by an appendix, comprising weights and measures, normal solutions, and an explanation of the metric system.

Having regard to the limited scope of this work, perhaps "Chemistry of the Urine" is too ambitious a title. Adverse criticism on this score, however, is in a

measure discounted by a statement in the preface to the effect that all mention of the methods of determining phosphates and most of the mineral constituents is omitted, *firstly*, because they are not of great pathological interest, and, *secondly*, because the author has nothing to say about them which cannot be found in every physician's and analyst's library. With the first of these reasons we entirely disagree, and the second, we hope, may speedily be remedied by Mr. Allen himself, who in his preface makes a promise that, if a second edition be called for, this and other omissions may be supplied.

That the determination of the phosphates in urine is of no pathological importance is, in our opinion, a mistake. It is a feature in the clinical study of the urine which promises a fruitful harvest. Among the many medical problems awaiting solution, and not unlikely to profit by a prosecution of the study of the urinary phosphates, are whole provinces of diseases of the nervous and osseous systems. An omission perhaps even less to be expected, from its close alliance with the subjects of diabetes and gout, is that of all detailed reference to oxaluria. The chemistry of the urine from its physiological side, as a rule, receives very scant attention. For example, in dealing with the quantity of urine passed by an average adult, no details are given, nor are the variations, or the causes of the variations, even alluded to. On the other hand, the physiological action of food upon urine, especially as affecting its reaction, is well described. In this connection, it is to be noticed that, when treating of the various adventitious odours of urine, no mention is made of the characteristic effect produced by the eating of asparagus.

The pathological sections, especially those dealing with diabetic and albuminous urines, are altogether admirable; they are thoroughly exhaustive, and so practically critical that it is difficult to find a single thing of importance that has escaped the author's keen observation. One small fact, however, may be mentioned; namely, a curious observation by the late Dr. Moxon, that the addition of nitric acid to urine passed by persons taking iodide of potassium produces an orange-coloured zone at the juncture of the two liquids—an appearance that is quite characteristic, but such, to one unaware of the fact, as might in some circumstances lead to confusion.

It is when we come to the purely practical details that we find our author at his best, as, for instance, when describing Knop's modification of the Kjeldahl process for the determination of nitrogen, or Gerrard's cyano-cupric process for glucose titration, or, again, his comparison and description of Fehling's and Pavy's processes and their modifications, together with the methods to be resorted to for the removal of disturbing elements in the determination of small quantities of sugar; in all of these and similar passages nothing could be more clear or satisfactory. In point of fact, from beginning to end the work is redolent of the laboratory, and will be found, alike by the clinical physician and technical chemist, a thoroughly practical and trustworthy guide—quite the best, so far as its scope goes, in the English language.

M. A. A.

APPOINTMENT.

Mr. GEORGE R. THOMPSON has been elected Public Analyst for Monmouthshire.

THE ANALYST.

DECEMBER, 1895.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday, November 6, at the Chemical Society's Rooms, Burlington House, at eight p.m., the chair being occupied by the President (Dr. Stevenson).

The minutes of the previous meeting were read and confirmed.

The following gentlemen were elected. As Members: Robert William Atkinson, B.Sc. (Lond.), F.I.C., and J. Walter Leather, Ph.D., F.I.C. As Associate: Herbert Burgess, assistant to Dr. Graham.

The following gentlemen were proposed for election as Members of the Society: Thomas Hawkins Percy Heriot, Chemist to Messrs. Welford and Sons, Limited; Frank H. Leeds, F.I.C., F.C.S., Analytical and Consulting Chemist; Alex. K. Miller, Ph.D., Analytical Chemist; C. A. Mitchell, B.A., A.I.C., Chemist to Messrs. Beaufoy and Co.; C. H. R. Moore, F.I.C., Chemist to the Sheppey Glue and Chemical Works, Limited; Clarence Arthur Seyler, B.Sc. (Lond.), F.I.C., Public Analyst for the County of Carmarthen, and Deputy Public Analyst for the County of Glamorgan and the Borough of Swansea; Benjamin Watmough, Analyst at Messrs. Brotherton and Co.'s Ammonia Works, Wakefield.

NOTE ON A RECENT MILK CASE INVOLVING A SAMPLE OF ABNORMAL MILK.

By R. BODMER.

On April 4 I received a sample of milk from one of my Sanitary Inspectors which yielded the following analytical figures:

Specific gravity	1028.10
Total solids	10.85 per cent.
Fat	3.20 "
Solids-not-fat	7.65 "
Ash	0.66 "

I returned it as containing 10 per cent. of added water.

The vendor sent his sample to another analyst, who reported it as containing 8 or 9 per cent. of added water. The vendor then had a certain cow in his shed (which contained about twenty cows) milked in the presence of his analyst, and the milk was found by him to be *equivalent* to one containing about 8 per cent. of

added water. The Inspector's sample was sent to Somerset House, and was reported to contain "not less than 10 per cent. of added water."

The case did not, however, stop here. The vendor appealed to the magistrate at Southwark Police Court to permit a particular cow to be milked in the presence of the Chief Sanitary Inspector for the Bermondsey Vestry. Permission was granted; but the "appeal to the cow" only came off on June 13, more than two months after the taking of the original sample. Two cows were milked separately, Nos. 1 and 2, and samples taken from each. I was not present at the milking, but cautioned the Inspector to see that the cows were milked dry.

The figures of the analyses were as follows :

Specific gravity	No. 1.	No. 2.
			1024·80	1028·90
Total solids	10·38 per cent.	11·68 per cent.
Fat	3·14	3·48
Solids-not-fat	7·24	8·20
Ash	0·88	0·84

Thus, on the 8·5 basis, No. 1. was equivalent to milk containing 15 per cent. of added water, and No. 2 to milk containing 3 per cent. of water.

The *ash* yielded by both milks was extremely high, whereas the ash yielded by the original sample was *low*, and quite in accordance with the presence of added water.

I reported the two samples as being abnormal. On the case being again heard, I gave evidence, and maintained that the original sample was a watered, and not an abnormal, milk. Dr. Dyer also appeared in support of my certificate.

The analyst of the vendor, however, stated that, although he had previously reported that the original milk was *watered*, he now believed it to be *genuine*. He altogether disagreed with Dr. Dyer and myself that the ash was any guide in discriminating between a watered and an abnormal milk, and that 0·66 ash was quite compatible with a genuine milk.

The vendor's wife and his stableman gave evidence that the original milk came from *one cow* (No. 1), and that no water was added.

The cows were, I believe, shorthorns, and were chiefly fed on brewers' grains.

The magistrate decided to dismiss the case, but declined to grant the defendant costs against the Vestry.

I also determined the *milk-sugar* in the No. 1 sample. The full analysis of No. 1 milk was, therefore :

Fat	3·14 per cent.
Milk-sugar	2·59
Casein	3·77
Ash	0·88

This bears out Mr. Richmond's theory that in abnormal milks the *milk-sugar* is generally deficient. Other observers have also noted the high ash in abnormal milks.

DISCUSSION.

Mr. CASSAL thought that the only satisfactory way of dealing with cases such as these, in which the adulteration was carried on "at the other end of the cow," by

improper feeding, with or without improper management, was to make it perfectly plain that the so-called milk was in reality not milk at all, but merely a pathological fluid which could not be regarded as coming within the meaning of the term "milk" as it was understood by the public, and as it was used by a purchaser under the Sale of Food and Drugs Acts. This had been pointed out repeatedly by the speaker and others at the meetings of the Society, in Courts of Law, and elsewhere, and it was to be regretted that the necessity for stating so obvious a fact should arise so frequently. The statement, which he gathered had actually been made in Court, that a proper determination of the ash of milk did not afford evidence in regard to the presence of extraneous water was, of course, absolutely incorrect.

Mr. WYNTER BLYTH thought it very extraordinary that a magistrate should have attached so much importance to evidence which was obtained after a two months' interval, and which he would have imagined had hardly any bearing at all upon the real question at issue.

Mr. RICHMOND thought that the cause of the unsuitability of brewers' grains for cattle feeding when given alone was probably due, not so much to the large proportion of water which they contained, as to the acidity which developed in them after they had been kept in stock for some time, and which caused severe diarrhoea.

Mr. HEHNER said that the matter of natural abnormality had nothing to do with the main question, which turned upon the actual composition of the article which a purchaser was entitled to expect when he asked for milk. This was really the only safe basis that could be adopted. No excuse whatever ought to be accepted for deficiencies (whether natural or artificial) from a reasonable and lenient standard of composition.

Dr. DYER remarked that in milk adulteration cases a difficulty sometimes arose as to the specific charge made against the defendant. If the charge was that the article sold was not "of the nature, substance and quality demanded," the case could be easily dealt with; but if the charge merely referred to the addition or abstraction of any ingredient, a magistrate seemed to be more or less bound to consider such a case in its strictly legal aspect, and if he was satisfied that the article had not been tampered with, but was really abnormal, it was difficult to see how he could convict on a specific charge of addition or abstraction.

In the case alluded to by Mr. Bodmer, a conviction would probably have been obtained, but for the statement made by the analyst who appeared for the defendant that there was nothing necessarily abnormal in a high or low ash. This analyst also stated that the percentage of ash obtained would depend to a great extent upon the method employed for determining it, losing sight, apparently, of the fact that the determinations had been in this case made under the same conditions by the same analyst, and confessing that he had himself not determined the ash.

Brewers' grains were used as a staple article of food by most large milk-producers, and, properly employed, must be considered to be one of the best artificial foods that could be given to cows. The fact of their being sour was not detrimental to their feeding value; in proof of this he instanced the case of a large farmer and supplier of milk to the London market, whose farm he frequently visited. This gentleman had a large pit, or silo, which he filled up with brewers' grains when these

were plentiful, keeping them pressed down for future use. They were often kept for months before they were consumed, being perfectly sour, but the milk produced was always of good quality. Grains, however, were not relied upon alone, but were supplemented by a reasonable quantity of concentrated food, viz., cake and meal. Thus supplemented, grains were exceedingly useful in promoting a good flow of milk, but, in order that the milk might be of good quality, an adequate supply of concentrated food, in addition to the highly watery grains and other moist, bulky food, was absolutely necessary.

The PRESIDENT remarked that saline matter was often added to grains in order to counteract their insipidity. He knew from observation on the human being that saline matters administered internally tended to appear in the milk secretion, and if this were also the case with cows, it might offer some clue to the cause of the high percentages of ash in these milks, if the assumption was correct as to brewers' grains being the main item in the diet.

Mr. BODMER said that he was of the same opinion as Mr. Cassal regarding the proper meaning of the term "milk." Every effort was made to explain the matter clearly to the magistrate, but the evidence of the defendant's wife and stableman appeared to create a doubt in his mind, the benefit of which was given to the defendant. He had brought the matter before the Society mainly for the sake of pointing out the high percentage of ash and low milk-sugar in the abnormal milk, in the hope that it might lead to the possibility of discriminating between milk which was really abnormal and milk which had been watered.

NOTE ON TWO SAMPLES OF "FILLED" OR LARD CHEESE.

By R. BODMER.

DURING the discussion on a recent paper on Cheese by Messrs. Chattaway, Pearmain and Moor, Mr. Allen remarked that so far no samples of "filled" or lard cheese had come under his notice in this country. I have recently analysed two such adulterated cheeses which gave the following figures:

	No. 271, July, 1895.	No. 347, September, 1895.
Fat	42.4 per cent. ...	31.7 per cent.
Valenta test	60° C....	70° C.
Reichert figure (2.5 grammes)	0.9 c.c. $\frac{N}{10}$ baryta	0.8 c.c. $\frac{N}{10}$ baryta.
Insoluble fatty acids	9.35 per cent.
I reported: No. 271, Skim-milk cheese	60 p.c. per cent.
Fat other than butter-fat	40 per cent.
No. 347, Skim-milk cheese	70 per cent.
Fat other than butter-fat	30 per cent.

Both samples were, I believe, American cheeses, and in both cases the vendors were convicted and fined.

THE COMPOSITION AND ANALYSIS OF CONDENSED MILK.

By T. H. PEARMAIN AND C. G. MOOR, M.A.

CONDENSED milk appears to have been first prepared about the year 1856, and is now an article of great importance, more particularly on account of the immense quantity used in the feeding of young children.

We may divide the various brands sold in this country into four classes. Many of these brands are the same milk under different names: 1, unsweetened milks; 2, sweetened milks; 3, sweetened partly-skimmed milks; 4, sweetened skimmed milks.

In most cases the degree of concentration is that obtained by evaporating three volumes to one; that is, the addition of two volumes of water (to an unsweetened milk) will produce a strength equal to the original.

1. The unsweetened milks, of which there are at present six different brands, are all well prepared, and keep perfectly. They contain the due proportion of fat.

2. This class forms by far the largest and most important part of the whole supply, and for the most part there is nothing to complain of in them, except that the degrees of dilution recommended would in every case produce a milk below standard. A few of them have been prepared from milk partly deprived of its fat.

3. Many of these which are stated to be prepared from milk from which a portion of its fat has been removed, are almost entirely devoid of fat, containing less than 10 per cent. of the original fat.

4. Separated milks (miscalled skimmed milks) are largely used by poor and ignorant people for infant feeding, and there can be no doubt but that great harm results from this practice.

It is now becoming more generally recognised that by the use of condensed milks all danger of milk epidemics is avoided, which is an additional reason for the favour they enjoy in the feeding of children.

In the appended table of analyses will be noticed a sample of Italian condensed milk; this is not sold in small tins for ordinary use, but only in bulk to dairymen for diluting down and selling as fresh cows' milk. It is admittedly partly deprived of its fat, but is supposed to be capable of producing four times its volume of ordinary milk.

In the *British Medical Journal*, July 27, 1895, is a report by Dr. Dyer on seventeen samples of condensed milk, nearly all of which were skimmed or partly-skimmed milks. In his report some of the labels were reproduced, which is a feature of great interest, as it has had the effect of causing a fresh issue of labels.

In the present state of the law as interpreted by the judicial authorities, condensed skimmed milk, that is to say, milk deprived of its fat, in the absence of which it ceases to be milk in the true sense of the word, may lawfully be labelled "Condensed Milk," provided the fact is printed somewhere on the label in more or less microscopical type that the tin "contains skimmed milk." In other words, a manufacturer may condense skimmed milk and describe it in large letters on the body of the tin as "Condensed Milk," whereas an ordinary milk-vendor is fined for selling skimmed milk, unless the fact is stated at the time of purchase. Attention has already been called to this point in the *British Medical Journal* report.

It is very probable that the high infant death-rate, particularly among the poor, is largely due to, if not actually caused by, the use of these skimmed condensed milks, which subjects the children to a process of slow starvation. In our opinion, it is not only very desirable that the words "Skimmed" or "Separated" should be printed on the label in as large and prominent letters as the words "Condensed Milk," but we would go further, and say that the statement "Skimmed milk is unfit for the nourishment of children" should be made compulsory. This

statement is made on the label of one brand of skimmed milk, and the manufacturers should not be compelled to suffer for their honesty in pointing out what less honest makers are ready to conceal. In the case of one brand of condensed skimmed milk, it is actually recommended for children's use. Even in the case of sweetened whole milks, the amount of water recommended to be added to the contents of the tin to produce a product for "ordinary" purposes or for infant feeding is in nearly every case largely in excess of what it should be, as will be seen from the following table of examples taken at random from the table of analyses.

Sweetened Whole Milk.	Dilution recommended for Household Purposes.	Percentage of Fat in such Product.	Dilution recommended for Infants' Use.	Percentage of Fat in such Product.
A	1 to 3	2.6	1 to 5	1.8
B	1 to 5	1.6	1 to 14	0.7
C	1 to 5	1.6	1 to 14	0.6
D	1 to 6	1.4	1 to 15	0.7
E	1 to 5	2.1	1 to 14	0.8
F	1 to 5	1.7	1 to 14	0.7
G	1 to 5	1.7	1 to 14	0.7
Human milk	—	—	—	3.0

In the above cases the extreme dilution recommended on the label is taken. Thus, it will be seen that the dilutions recommended for children's use, except in one case, contain on the average less than one quarter the amount of fat they should contain when compared with human milk.

THE ANALYSIS OF CONDENSED MILK.

The contents of the tin are well mixed, and 10 grammes weighed into a beaker and made up to 100 c.c. We now have a 10 per cent. solution, which serves for the following estimations:

Total Solids.—20 c.c. of the solution are evaporated in a platinum dish. The milk solids take at least six hours to dry to constant weight.

Ash.—This is determined on the above by igniting at as low a temperature as possible.

Proteids.—10 c.c. of the solution are evaporated to dryness in a flask, and the nitrogen determined by the Kjeldahl process. The proteids are then calculated by the usual factor.

Milk-sugar.—10 c.c. of the solution are made up to 100 c.c. by the addition of 40 c.c. of water and 50 c.c. of ammonia. The milk-sugar is then determined in this 1 per cent. solution by Pavy's method.

Fat.—Two quantities of 5 c.c. of the solution are placed on two Adams' papers, well dried, and the coils extracted with anhydrous ether.

It is well known that the Werner-Schmidt process cannot be applied to condensed milks on account of the solubility of caramel in moist ether, which renders the Adams process the only accurate method available for the estimation of the fat. In view of the length of time taken by this process, we have made repeated attempts to determine the fat by the Leffmann-Beam machine. This, we are satisfied, can be

done with a fair amount of accuracy if the following details are adhered to in every respect, special attention being paid to the strength of the sulphuric acid. The great objection to this method is the large factor that has to be employed, namely, 15·5, seeing that we find it necessary to work on only 1 grammé of the original sample. The procedure is as follows: 10 c.c. of the 10 per cent. solution of the sample are run into the bottle, and 3 c.c. of the hydrochloric acid-fusel-oil mixture added, the bottle well shaken, and then 15 c.c. of sulphuric acid of 85·0 per cent. added with agitation. Sufficient of a hot mixture of sulphuric acid and water (1:2) is then added to the bottle to bring the top of the liquid nearly up to the zero mark. The bottle is then "whirled" in the machine for three minutes. The fat will not all come up at once, but after placing the bottle in the water-oven for two or three minutes, and again "whirling," the entire amount of fat will be obtained, which is then carefully measured off with a pair of dividers.

Respecting the relations of the fat, proteids and milk-sugar in condensed milk, it does not seem to be so constant as might be expected. We note a curious difference in the relations of these bodies in the unsweetened condensed milks we have examined when compared with some analyses published by Battershall ("Food Adulteration and its Detection," p. 53). These analyses show the fat, proteids and milk-sugar to be nearly equal, whereas it is customary to find these constituents to be in the ratio of 2 : 2 : 3.

In conclusion, we hope that the recent agitation in connection with fraudulent condensed milk will call attention to what may be termed, without exaggeration, a national question.

ANALYSES OF CONDENSED MILKS.

Brand.	Total Solids.	Fat.	Milk-sugar.	Pro-teids.	Ash.	Cane-sugar (by difference).	Description on Label on Tin.
Anglo-Swiss ...	74·4	10·8	16·0	8·8	1·7	37·1	Unskimmed.
Beehive ...	77·7	0·2	—	—	2·6	—	Skimmed.
Calf ...	58·0	1·0	16·0	7·5	1·6	31·9	Skimmed.
Cleaves ...	71·0	10·8	17·1	10·1	1·7	31·3	Not skimmed.
Clover-leaf ...	76·0	10·7	13·6	8·8	2·0	40·9	Guaranteed to contain all its original cream.
Cow ...	74·9	2·0	13·0	11·5	2·6	45·8	Partly skimmed.
Cowslip ...	70·9	1·4	14·6	11·4	1·6	41·9	Skimmed; guaranteed to be entirely pure.
Cradle ...	29·9	9·5	15·5	3·1	1·8	none	Humanized condensed milk.
Cross ...	75·0	1·2	16·0	10·5	2·6	44·7	Skimmed.
Cup ...	56·9	1·0	15·4	8·5	1·6	30·4	Skimmed.
Daily ...	68·8	1·3	13·7	10·2	2·5	41·1	Skimmed.
Daisy ...	64·0	0·5	—	—	2·2	—	Skimmed.
Darby and Joan ...	73·1	9·8	13·0	13·3	2·3	34·7	Contains nothing but full cream milk.
Drummer-boy ...	—	1·0	—	9·6	—	—	Skimmed.
Farmhouse ...	77·0	0·4	—	—	1·7	—	Skimmed.

ANALYSES OF CONDENSED MILK—*continued.*

Brand.	Total solids.	Fat.	Milk-sugar.	Pro- teids.	Ash	Cane- sugar (by differ- ence).	Description on Label on Tin.
Favourite ...	70.6	0.3	—	10.0	2.1	—	Skimmed.
Fern ...	67.7	10.7	15.0	10.6	1.6	29.8	Quite genuine.
First Swiss ...	36.7	10.5	14.2	9.7	2.1	none	Unsweetened; guaranteed pure and unskimmed.
Fourpenny ...	76.5	10.4	13.0	9.8	2.0	41.3	Not skimmed.
Full Weight ...	76.5	11.0	13.5	12.3	2.5	37.2	Not skimmed.
Geranium ...	75.0	9.8	13.0	7.5	1.6	43.1	Guaranteed perfectly pure.
Go-a-head ...	76.1	10.0	14.6	9.7	2.1	39.7	Guaranteed pure; no part of the cream has been extracted.
Goat ...	71.0	1.2	12.0	9.9	2.0	45.9	Skimmed.
Golden Eagle ...	—	1.0	—	6.8	—	—	Guaranteed pure milk, from which a portion of the cream has been extracted.
Gowan ...	72.0	10.8	13.4	10.5	1.7	35.6	Partly skimmed.
Handy ...	75.5	0.3	17.0	12.3	1.6	44.3	Skimmed.
Hollandia ...	43.0	9.8	18.5	11.3	2.5	none	Best quality; without sugar.
Home ...	71.3	1.3	12.5	11.1	2.5	43.9	Skimmed.
Home and Colonial ...	72.6	13.5	17.0	9.7	1.9	30.5	(No description on label.)
Household ...	70.0	0.3	—	—	2.0	—	Separated milk.
Ideal ...	38.0	12.4	16.0	8.3	1.7	none	Enriched with 20 per cent. of added cream (not sweetened).
Imperial Dairy ...	70.4	3.7	12.6	11.3	1.6	41.2	Skimmed.
Italian ...	44.6	9.5	16.5	14.7	3.5	none	Unsweetened; sold for diluting down.
Lancer ...	67.6	0.3	16.6	12.3	2.6	35.8	Skimmed.
Lifeguard ...	65.8	0.3	—	—	2.4	—	Skimmed.
Lipton's ...	71.0	9.3	14.5	7.9	2.2	37.1	Prepared from the richest pure milk.
Lovers ...	73.0	0.2	—	—	2.1	—	Skimmed.
Lucerne Lion ...	71.7	10.8	15.2	9.3	2.0	34.4	Guaranteed finest quality (mountain milk).
Minstrel ...	75.3	0.2	15.4	9.7	1.7	48.3	Skimmed.
Mother ...	72.0	8.8	13.7	7.3	1.7	40.5	Guaranteed to be prepared with the best and richest cows' milk.
Milkmaid ...	76.3	11.0	14.6	9.7	2.3	38.7	Swiss; genuine.
Nestlé's ...	77.2	13.7	15.0	9.7	1.6	37.2	Prepared from pure milk of Swiss cows.
Rose ...	76.6	12.4	17.6	8.3	2.2	36.1	Not skimmed.
Scandinavian ...	74.6	10.8	14.5	6.6	1.6	41.1	Perfectly pure.
Shamrock ...	71.6	0.5	18.4	11.8	2.0	38.9	Skimmed.
Springtime ...	74.0	0.3	—	—	2.2	—	Skimmed.
Tip-top ...	74.0	10.7	15.4	8.8	1.7	37.4	Not skimmed.
Viking ...	34.2	10.0	13.3	9.0	1.9	none	Unsweetened (full cream).
Wasp ...	73.4	1.0	12.3	11.0	1.9	47.2	Skimmed.
World's Tea Co. ...	75.0	9.3	—	—	2.0	—	Full cream.

DISCUSSION.

Dr. DYER said that, as some recently-published analyses made by him of condensed milk had been referred to, he would like to say that he had nothing to do with the selection of the various brands analysed, and really did not know how far they consisted of brands having a free sale, and how far of less common brands. He did not purchase the samples, but received them from the editor of the *British Medical Journal*, with an instruction to determine the fat and report the result.

Mr. RICHMOND said that many of the percentages of ash given in the table were distinctly lower than those which he had been accustomed to regard as normal to milk. Dr. Vieth had found that the amount of ash in milk was almost invariably about 8.3 per cent. of the solids-not-fat, and experiments which he (the speaker) had himself made completely confirmed the correctness of this proportion.

Mr. BEVAN said he would like to ask the authors if they were quite satisfied with the Adams method for fat determination. It seemed to him that in condensed milk the conditions were far from favourable to complete extraction of the fat.

Dr. DUPRÉ, referring to the statement made by the authors that condensed milk was free from all risk of imparting infectious diseases, said that he remembered a case in which an outbreak of scarlatina was distinctly traceable to some condensed milk which had been used, and in which Dr. Klein detected the micrococcus of scarlatina.

Mr. HEHNER remarked that all the milks referred to in the paper seemed to be correctly described as whole milks or skimmed milks on the labels. It was very important, however, to draw attention to the directions given as to the addition of water, which were in most cases entirely wrong. The "condensed milkman" (if the term might be used) appeared to be in an unfortunate position, since the addition of the proper proportion of water—namely, about 2 parts of water by weight to 1 of milk—resulted in a product which was an almost undrinkable syrup; while, if a sufficient quantity was added to make it palatable, the nitrogenous matters were reduced to a minimum. As a matter of fact, it was impossible to make from condensed milk anything that could be said to correspond with the original milk, and attempts to do so ought to be entirely discouraged. At the same time, the manufacture of skimmed or separated condensed milk was, in his opinion, a perfectly legitimate trade, and if the article was properly and plainly labelled, no normal objection could be raised against it. The proportions of ash found by the authors of the paper seemed to vary a great deal. He noticed one instance in which the proportion was 3.5 per cent., which would mean that the original milk, containing, say, 0.7 per cent., was condensed five times, provided that no other ash had been added. Others contained as little as 1.4 per cent. He could not help thinking that some error must have crept into these determinations.

The PRESIDENT remarked that, taking the average of the last twelve milks in Messrs. Pearmain and Moor's table, which were all that he had had time to critically examine, the ratio which the ash bore to the solids-not-fat agreed fairly well with the normal proportion. The average ratio between the ash and the proteids was also almost exactly normal. In fact, these last twelve cases seemed to bear out the assumption that the analyses had been fairly and correctly made.

Mr. MOOR, in reply to Mr. Bevan's question regarding the Adams process, said it was the most satisfactory method they had been able to find. They had tried the Leffmann-Beam process in eight or nine cases, and found it to work well, even when a large quantity of cane sugar was present, if the strength of the acid was strictly adhered to. As, however, the estimation could be made in about five minutes, this method seemed to be worthy of a more extended trial. In working the Adams process, it was their practice, after the extraction was finished, to dry the coil and go through the operation of extraction a second time, so as to make sure that all the fat was obtained. With regard to the question of disease propagation, the thermal death-point of nearly all disease organisms ranged between 50° and 60° C., and as the process of manufacture involved the raising of the milk to a temperature of 70° C. four successive times, it did not seem probable that any disease organisms could survive.

Dr. DUPRÉ pointed out that, although all mature disease organisms would be killed by the heat, spores of some of these organisms might still survive and develop in the condensed milk after the processes of manufacture had been completed.

Papers were read on the following subjects, the publication of which is unavoidably deferred: "Note on the Estimation of Minute Quantities of Metals in Liquids," by E. Russell Budden and H. Hardy; "Note on a convenient form of Polarimeter for examining Essential Oils," by E. Russell Budden.

NOTES ON COMMERCIAL CONDENSED MILKS.

BY ALFRED H. ALLEN.

IN the summer of 1894 I received, under the Sale of Food and Drugs Act, a fairly complete collection of the brands of condensed milk then in the market. The results obtained are interesting in connection with the paper of Messrs. Moor and Pearmain, as they show the change in practice of manufacturers of condensed milk since that date. The partially-skimmed milks which figure so largely in the table of results appended to this paper have now nearly disappeared from commerce, and it will be noted that the character of several of the brands analysed by Messrs. Moor and Pearmain and also by myself has materially changed. Another practice which it is well to bear in mind is that if, through proceedings in court or other circumstances, the quality of a brand of condensed milk is challenged, certain manufacturers simply rechristen the product, and reissue the old tins as some other brand.

As the proportion of fat in unskimmed milk is fully equal to, or somewhat in excess of, the proteids, a comparison of these two constituents affords a ready means of ascertaining whether any notable quantity of the cream natural to the milk has been removed.

With regard to the general character of condensed milk, while some few manufacturers prepare a first-rate article, for which they obtain a first-rate price, in other cases the statements made on the label are by no means borne out by the analyses. Particularly objectionable are the statements with respect to the amount of dilution

to be practised in order to restore the milk to its original concentration. As the amount of condensation is never more than 3 to 1, directions to dilute the milk with 6 to 8 parts of water, and for special purposes to add from 10 to 15 parts of water, are open to grave animadversion. It seems to be the case, however, that, as the law at present stands, there is no penalty under the Sale of Food and Drugs Act for attaching to an article a label bearing untrue statements or misleading directions. The practice might be dealt with under the Merchandise Marks Act, but with that enactment public analysts are not officially concerned.

Brand.	Total Solids.	Fat.	Proteids.	Description on Label.
Alderney ...	68.10	11.05	10.95	Guaranteed to contain 60 per cent. of original cream.
Anglo-Swiss ...	73.70	9.70	9.87	Best unskimmed country milk.
Arcadia ...	71.20	8.08	10.25	From best and purest cow's milk.
Calf ...	66.30	—	10.19	Contains skimmed milk.
Cow ...	72.50	6.50	10.57	From partly-skimmed milk.
Cowslip ...	72.10	0.18	10.20	Skimmed milk.
Daily ...	69.64	0.26	10.58	From skimmed milk.
Devon ...	70.60	8.50	10.63	From skimmed milk.
Farm ...	66.60	0.12	10.14	From skimmed milk.
First Swiss ...	28.37	8.76	10.14	Unsweetened.
First Swiss ...	36.10	11.06	12.75	Unsweetened.
Fourpenny ...	75.36	5.40	13.18	From pure fresh milk containing all its cream.
Full weight ...	75.70	11.60	11.27	Warranted not skimmed.
Geranium ...	73.98	10.35	8.56	Full cream.
Goat ...	79.10	4.30	10.44	From skimmed milk.
Handy ...	67.38	0.17	10.20	From skimmed milk.
Home ...	69.44	0.91	9.13	From skimmed milk.
Milkman ...	73.66	11.80	11.40	Warranted to contain all original cream.
Mother ...	70.60	5.57	8.32	From unskimmed milk.
Popular ...	71.75	2.47	10.14	From partly-skimmed milk.
Rose ...	72.40	10.30	9.75	Warranted not skimmed.
Shamrock ...	71.30	—	10.88	From skimmed milk.
St. Olaf ...	71.86	11.30	10.50	From pure unskimmed milk.
Sunlight ...	71.76	5.60	8.63	From unskimmed milk.
Swiss (Nestlé's) ...	75.00	13.50	10.44	From pure milk, and only small quantity of pure cane-sugar.
Swiss (Nestlé's) ...	70.41	11.03	10.51	From pure milk, and only small quantity of pure cane-sugar.
Threepenny ...	66.25	0.30	10.49	From partly-skimmed milk.
Tip-top ...	74.25	8.12	8.82	Warranted not skimmed.
Viking ...	35.16	10.40	9.14	Unsweetened.

NOTE ON THE IODINE AND BROMINE ABSORPTIONS OF
LINSEED-OIL.

BY ROWLAND WILLIAMS.

Iodine Absorption.—The percentage of iodine absorbed by raw linseed-oil, as recorded by different observers, varies considerably, and is, according to my experience, a good deal too low. Thus, in Allen's "Commercial Organic Analysis," vol. ii., p. 50, the original figures of Hübl are quoted as 156 to 160 per cent., whilst Alder Wright ("Oils, Fats, Waxes," p. 183) states the average of a large number of results, obtained by numerous observers and collated by Benedikt, to be 175 per cent.

Allen, in his article on "Fixed Oils and Fats" (Thorpe's "Dictionary of Applied Chemistry," vol. iii., p. 29), gives the iodine absorption as 154 to 160 per cent., although in another part of the same article, while repeating these figures, he mentions the fact that Thomson and Ballantyne (*Journal of the Society of Chemical Industry*, vol. x., p. 233) found considerably higher percentages. The two last-named chemists are, I think, the only observers who have recorded what I have every reason to believe the correct iodine number.

During the last four years I have had exceptional opportunities for the examination of samples representing large bulks of raw linseed-oil (several hundred samples having passed through my hands during that period). Of these, none which I believe to be genuine absorbed less than 180 per cent. of iodine, whilst in the majority of cases the number was nearer 190 per cent., and occasionally exceeded that figure.

I should explain that these figures refer mainly to Baltic linseed-oil, but I have also always found the iodine number of other kinds of linseed-oil (*e.g.*, Indian and River Plate) to be much higher than that usually recorded. Taking at random from my note-book the results of fifty-three samples of Baltic oil, I find the average iodine absorption to be 188.5, and that of six samples of other varieties to be 183.3 per cent. In my determinations I always add a large excess of Hübl's reagent (usually at least double the amount likely to be absorbed), and allow to stand overnight (eighteen to twenty hours), and it is to these two points that I attribute the discrepancy between my figures and those of most other observers. If only a small excess of iodine be employed, and the reaction be permitted to proceed for only four to six hours, as is, I believe, frequently the case, the results obtained will certainly be too low.

Bromine Absorption.—Previously to the appearance of Mr. Hehner's paper on "The Determination of the Bromine Absorption of Fats gravimetrically" (*THE ANALYST*, vol. xx., p. 49), I had not worked in this direction, but since then I have made a large number of bromine absorption determinations, working on the lines suggested by Mr. Hehner. Having convinced myself of the accuracy of this gravimetric method, especially as regards raw linseed-oil, I can confidently recommend it as being in many cases both more convenient and more reliable than the volumetric iodine estimation. It possesses the undoubted advantage of leaving the haloid product in a state suitable for further examination of its physical and other characters if desired. When determinations of the halogen absorptions of oils and fats have to

be made only comparatively rarely, the gravimetric bromine method is certainly to be preferred, as, after one blank experiment to ascertain the freedom of the bromine and chloroform from fixed residue, these reagents are ready for use at a moment's notice. On the other hand, it is essential that Hübl's solution should be fairly newly made, and yet not too new (it is advisable to prepare it at least several hours previous to use), otherwise irregular results will most probably ensue.

Boiled Linseed-oil.—The bromine absorptions of linseed-oil, both boiled and raw (102.4 per cent. and 76 per cent. respectively), as recorded by Mills (Allen's "Commercial Organic Analysis," vol. ii., p. 50), are so obviously wrong as hardly to need correction. Still, it may be interesting to place on record my own observations on this point. It must, however, be remembered that Mills's results were obtained by a colorimetric volumetric process, whereas my own bromine determinations are all gravimetric; so it is perhaps scarcely fair to compare the figures.

On *a priori* grounds one would naturally expect that the thicker the consistency to which linseed-oil has been boiled, viz., the farther removed from its original raw condition, the lower will be its bromine absorption, and *vice versa*. Being in a position where I have special facilities for making gravimetric determinations of the bromine absorption of oil at various stages of boiling on the large scale, I am able to say that this theory always holds good.

In the following table I have recorded the iodine (volumetric) and bromine (gravimetric) absorption figures of a number of samples of linseed-oil, both raw and at different stages of boiling:

	RAW.						BOILED.			
	1.	2.	3.	4.	5.	6.	Thin.	Thin.	Stout.	Very Stout.
Iodine	183.2%	192.9%	185.2%	195.5%	194.8%	195.1%	175.1%	168.0%	99.5%	96.9%
Bromine	114.2%	120.7%	115.1%	119.2%	119.6%	119.4%	111.3%	112.4%	65.6%	59.9%

It will be observed that the ratio between the iodine and bromine figures of raw linseed-oil is very regular, being on the average as 1.62 to 1 (theoretically it should be 1.5875), but in the case of boiled oil the ratio is somewhat irregular.

Examination of Butter. E. Polenske. (*Arch. Gesundheitsw.* ii., 523; through *Zeit. angew. Chem.*, 1895, pp. 581, 582.)—This method is based on the greater solubility of butter-fat in absolute alcohol than of the foreign fats used as adulterants. Twenty-five grammes of the clear butter-fat are weighed into a dry 250 c.c. Erlenmeyer flask, and 125 c.c. of neutral absolute alcohol (specific gravity 0.7963 at 15° C.) added. On warming to about 50° C., and gently shaking, the fat readily dissolves. The flask is then placed in a water-bath maintained at 18° C., being at first loosely corked, but afterwards, when the contents of the flask are also at 18° C., well corked. After an hour the flask is well shaken, the agitation being repeated about ten times at intervals of three minutes. After the contents have stood at 18° C. for one and a half hours in all, 118 c.c. of the alcoholic solution are rapidly filtered through a paper of 10 c.m. diameter, precautions being taken to maintain the temperature as nearly as possible at 18°. The insoluble residue is melted, brought into a flat dish, and, after evaporation of the alcohol, the residue dried for three hours at 100° C. This gives the solid portion of the butter-fat for the investigation. Ten c.c. of the 118 c.c. of the filtrate

are evaporated in a weighed flask, and the weight of the residue multiplied by 11.8 gives the amount of the fluid portion of the butter-fat. The remainder of the filtrate is evaporated, dried, and separately examined, as in the case of the solid fat.

For this method of separation the butter-fat should be as fresh as possible. Five grammes of fat in the fresh condition were neutralized by 1.2 c.c. $\frac{N}{10}$ potash. After being kept three weeks in a cool place there was no appreciable alteration; but after two months the amount of alkali required was 1.8 c.c. At the same time, the solubility in alcohol had risen from 36 to 38.2 per cent.

From the examination of fifty samples of summer and winter butter, the author finds:

1. The limits of the Reichert-Meissl number are closer in the two portions of the fat than in the entire untreated fat. Thus, while with butter-fat the limits are about 8 c.c. apart, in the solid portion this is narrowed to 6 c.c., and in the fluid portion to 5.9 c.c.

2. With a low Reichert-Meissl number the solubility of the fat in alcohol is less.

3. A slight solubility of the fat in alcohol is not always associated with a lower Reichert-Meissl number of the fluid portion.

Experiments made with mixtures of butter with various foreign fats show that by taking the ordinary constants on the fluid portion of the fat more valuable results are obtained than when they are determined on the whole fat. C. A. M.

Acidimetric Estimation of Vegetable Alkaloids. L. F. Kebler. (*Journ. Amer. Chem. Soc.*, xvii., 1895, pp. 822-831.)—This paper is a study of the value of different indicators in the estimation of alkaloids by titration. In the author's opinion, the discordance in the results of different chemists may be largely attributed to the personal equation, each worker arbitrarily assuming his own end-reaction tint. In these experiments the titrations were made from acid to alkaline solutions, and the tints taken for the end-reactions were: Brazil-wood from yellow to onion-red, the purple ultimately fading to this; cochineal from yellow to bluish-red; hæmatoxylin from yellow to brown-orange; litmus from red to onion-red, and methyl-orange from red to straw-yellow.

After testing the value of these by titrating standard acid against a standard alkali, the titration of commercially pure alkaloids was undertaken. In the case of quinine and codeine, 2 grammes of the alkaloid were dissolved in alcohol in a cylinder, and the solution made up to 100 c.c. with alcohol. To 10 c.c. of this, after the addition of the indicator, decinormal acid was added in slight excess, the liquid well agitated, and the excess of acid titrated back with decinormal alkali.

Where the alkaloid was insoluble in alcohol, the two grammes were warmed in a beaker on the water-bath with 75 c.c. of decinormal acid, until the alkaloid dissolved, the solution being then cooled and made up to 100 c.c. with water. Each 10 c.c. then contained 0.2 of a gramme of alkaloid and $7\frac{1}{2}$ c.c. of decinormal acid solution. After adding the requisite amount of indicator to 10 c.c. and diluting to 50 c.c., the excess of acid was determined. Every precaution was taken in preparing the in-

dicators, and the following quantities were used in each case: Cochineal and litmus prepared as described in Sutton's "Volumetric Analysis," the former 5 drops, the latter 10; phenolphthalein, 1 gramme per litre of 50 per cent. alcohol, 5 drops; hæmatoxylin, 1 gramme in 100 c.c. strong alcohol, 3 drops; Brazil-wood solution, 5 drops; methyl-orange, 1 gramme in a litre of distilled water, 5 drops.

The results obtained with several pure alkaloids were:

Indicators.	Quinine.		Strychnine.	Morphine.	Codeine.
	La Wall.	Kebler.			
Brazil-wood ...	99.90	101.97	99.36	98.93	95.75
Cochineal ...	105.56	102.54	103.20	99.08	97.09
Hæmatoxylin ...	99.81	103.37	100.03	98.17	95.90
Litmus ...	101.80	103.55	103.54	98.93	96.38
Methyl-orange ...	—	103.27	104.21	100.59	98.11

The figures in the first column are those obtained by an independent worker.

The following table shows the applicability of the process to crude alkaloids:

Indicators.	Crude Morphine.		Crude Cocaine.
	La Wall.	Kebler.	
Brazil-wood ...	99.23	98.47	95.90
Cochineal ...	100.14	99.53	97.11
Hæmatoxylin ...	99.08	97.59	95.74
Litmus ...	99.50	98.93	96.82
Methyl-orange ...	102.10	100.02	100.14

With the same crude morphine the ash method gave 97.59 per cent., the lime-water method 98.22 per cent., and the absolute alcohol method 98.33 per cent. of pure morphine. The crude cocaine yielded, by the gravimetric method of Dr. Squibb (*Ephemeris*, iii., 1171), 97.3 per cent. of nearly pure cocaine.

As it is often necessary to extract the alkaloids from their natural sources, the author has employed a modification of Keller's process for the purpose. To 10 grammes of the dry drug in a 250 c.c. flask, 25 grammes of chloroform and 75 grammes of ether are added, the flask well corked and shaken for some minutes. Ten grammes of 10 per cent. ammonia-water are then added, and the shaking continued at intervals for an hour. On adding 5 grammes more of the ammonia-water, the suspended powder coagulates, and the liquid can be poured off almost completely.

(1) Fifty grammes are evaporated on the water-bath, 10 c.c. of ether added, and again evaporated. The residue is dissolved in 15 c.c. hot alcohol, and water added to slight permanent turbidity. The indicator is then added and an excess of the standard acid solution, which is titrated back with centinormal alkali.

(2) Fifty grammes are shaken with 20 c.c. of acidulated water in a separating funnel, the aqueous solution removed to a second separating funnel, and the shaking repeated twice more with 15 c.c. of slightly acidulated water. The acidulated water in the second funnel is made alkaline with ammonia, and the alkaloid removed successively with 20 c.c., 15 c.c., and 15 c.c. of a mixture of three parts (by volume) of chloroform and one of ether. The residue left on evaporation of the solvents is then treated as in (1) above.

Nux vomica and *ipecacuanha*-root were treated according to process (1) and (2); *belladonna*-leaves according to (2). The results were as follows :

	Per Cent. Alkaloids in <i>Nux Vomica</i> by Process (1).		Per Cent. Alkaloids in <i>Nux Vomica</i> by Process (2). Gravimetrically.		Per Cent. Alkaloids in <i>Nux Vomica</i> by Process (2). Volumetrically.		Per Cent. Alkaloid in <i>Ipecac-root</i> by Process (1).	
	La Wall.	Kebler.	La Wall.	Kebler.	La Wall.	Kebler.	La Wall.	Kebler.
Brazil-wood...	2.04	2.58	2.94	3.00	2.37	2.37	2.46	2.54
Cochineal ...	2.64	2.69	2.86	3.10	2.42	2.39	2.59	2.49
Hæmatoxylin	2.18	2.24	2.88	3.11	2.23	2.27	2.48	2.54
Litmus ...	2.38	2.34	2.93	3.05	2.55	2.37	2.55	2.57
Methyl orange	3.02	3.64	2.93	3.02	2.65	2.61	2.95	3.30

	Per Cent. Alkaloid in <i>Ipecac-root</i> by Process (2). Gravimetrically.		Per Cent. Alkaloid in <i>Ipecac-root</i> by Process (2). Volumetrically.		Per Cent. Alkaloids in <i>Belladonna-leaves</i> by Process (2). Gravimetrically.		Per Cent. Alkaloids in <i>Belladonna-leaves</i> by Process (2). Volumetrically.	
	La Wall.	Kebler.	La Wall.	Kebler.	La Wall.	Kebler.	La Wall.	Kebler.
Brazil-wood...	2.58	2.60	2.36	2.35	0.26	0.20	0.19	0.15
Cochineal ...	2.63	2.68	2.52	2.33	0.28	0.20	0.24	0.14
Hæmatoxylin	2.58	2.68	2.35	2.33	0.27	0.22	0.21	0.13
Litmus ...	2.62	2.60	2.40	2.25	0.24	0.18	0.20	0.15
Methyl-orange	2.66	2.63	2.89	2.61	0.25	0.20	0.23	0.20

The *nux vomica*, examined by the method of Dunstan and Short, showed 2.89 per cent. of crude alkaloid, and this, titrated with acid solution, yielded 2.12 per cent. of pure alkaloid. The figures above show that the Keller process produces an alkaloid residue containing a larger percentage of pure alkaloid.

The conclusions arrived at with regard to the use of the different indicators are :

1. Methyl-orange is unsatisfactory with all strengths of acid, and litmus as ordinarily prepared is also unsuitable.

2. Hæmatoxylin, Brazil-wood, and cochineal give very promising results, hæmatoxylin being the best, and Brazil-wood the next best.

C. A. M.

The Theory of Hübl's Iodine Process, and a Suggestion for its Improvement.
Waller. (*Chem. Zeit.*, 1895, xix., 1786 and 1831.)—The author confirms Hübl's statement that iodine by itself is not completely absorbed by fats. A sample of cotton-oil, heated for an hour to 150° C. with an excess of the reagent, absorbed only 66.6 per cent. The action of nascent iodine is also incomplete. In alcoholic solution the quantity taken up depends on the concentration: one sample of oleic acid (iodine number 93) absorbed 43 per cent. from a 5 per cent. solution, 45 per cent. from one of 10 per cent., 76 per cent. from one of 20 per cent., 78 per cent. from one of 40 per cent., and 82 per cent. from one of 100 per cent. strength. (The presence of the oleic acid increases the solubility of the iodine in alcohol, thus accounting for the possibility of using the two latter solutions.) From 10, 20, and 40 per cent. chloroform solutions, the same oleic acid took up 53, 59, and 59 per cent. of iodine respectively. From 20 per cent. ethereal solution it absorbed 37 per cent., and from a similar solution in carbon bisulphide the absorption was 56 per cent.

A sample of oleic acid (prepared from olive-oil, iodine number 91), treated with

excess of pure bromine, gained in weight an amount equivalent to an iodine number of 95.9. Olive-oil (iodine number 81.55), after being treated twice, gave a number equal to 90.5. An older sample, whose value had fallen from 82 to 73, absorbed the bromine to an amount corresponding to an iodine number of 95.3. It would seem therefore as if the degree of oxidation of an oil might be determined in this manner. The following experiment shows that these high figures obtained on using bromine are not due to substitution. One gramme of olive-oil (iodine number 81.55) was treated for half an hour with 10 c.c. of an 8 per cent. solution of bromine in chloroform; the absorption of bromine was equivalent to 81.45 of iodine. The employment of a solution of potassium bromate and bromide is inconvenient, owing to the lack of a solvent that will withstand the action of the nascent bromine. It might be possible, perhaps, by determining the total bromine used, and also the amount of acid set free during the reaction—converting the latter into bromine, and subtracting it from the total halogen—to attain the desired result.

Experiments were tried with iodine trichloride. Two grammes of oleic acid (iodine number 91) were treated with 1.5 grammes of the chloride; the excess of the halogens was removed, the oil saponified, and the soap ignited. Analysis of the residues gave an iodine number of 94.8.

On an investigation of Hübl's process as ordinarily carried out, it was found that at the end of the operation the whole of the oil was in the chloroform solution, that the chloroform contained both iodine and chlorine, and that the fat contained both halogens in fairly constant quantity. The whole of the mercury (as double iodide) was in the alcoholic layer, and during the absorption free hydrochloric acid was found to be produced, the latter decreasing in amount with the age of the solution, but increasing in proportion to the excess of mercuric chloride used. To this free acid is due the fact that the ordinary iodine number does not correctly express the amount of iodine and chlorine taken up by the fat. It would seem that the nascent chlorine is in part prevented by this free acid from combining with the oil. One gramme of oleic acid (iodine number 91) was treated with the mixed alcoholic solutions of 0.9 gramme of iodine, and 1.08 grammes of mercuric chloride. After decolorization, the liquid was evaporated at 25° to 30° C. to 10 or 15 c.c., till hydrochloric acid vapours appeared. It was diluted with water, extracted with 30 c.c. of chloroform, and thrown on a tared filter. In the filtrate the acid was 0.133 gramme of HCl, or 0.129 gramme of chlorine. The chloroform solution, which contained the greater part of the mercuric iodide precipitated by the water, was passed through the same filter, and the weight of the iodide found to be 1.18 grammes, corresponding to 0.66 gramme of iodine and 0.52 gramme of mercury. From the aqueous liquid, on addition of sulphuretted hydrogen, there was obtained 0.283 gramme of mercury, 0.04 gramme of iodine, and 0.212 gramme of chlorine. The weight of the fatty residue was 1.24 grammes, and contained 0.18 gramme of iodine and 0.057 gramme of chlorine.

The absorption of the halogens by the oil apparently takes place as follows: Some of the iodine is absorbed, and the mercuric chloride is converted into iodide and free chlorine. The water in the alcohol prevents the latter from being taken up by the oil, hence hydrochloric acid is formed and oxygen set free. This then partly combines with that portion of the fat not yet attacked by the halogens, and renders

further action impossible. It follows, from the above, that for every 2 parts of iodine only 1 of mercuric chloride takes part in the reaction. That acid is set free during the process may be proved by comparative titration of the solutions, after decolorization with thiosulphate, by means of decinormal soda and litmus before and after the action on the oil. The amount of acid liberated, even when working on the same fat, is not constant: it varies with the age of the iodine solution, whether the iodine and the mercury are added together or separately, and on the excess of mercury employed. One sample of oleic acid (iodine number 93) gave, with forty-eight days-old iodine, the number 91.65, the free acid being equal to 0.0762 gramme of iodine. In order, therefore, to obtain the *true* iodine number, the latter amount must be subtracted, and the value thus becomes $(91.65 - 7.62) 84$. The same fatty acid, treated with 0.5 gramme of iodine and 0.48 gramme of mercuric chloride in perfectly fresh solution, gave $(92.1 - 13.9) 78.2$. (As a check, the halogens in the addition product were determined, and, reckoned as iodine, gave a value of 78.6.) Other tests with increasing amounts of mercuric chloride, the iodine remaining the same, gave lower figures: (1) Using 0.5 gramme of chloride gave $(92.65 - 16.45) 76.2$, and (2) using 2.0 grammes of chloride $(96 - 24)$, 72.

The falling off of the strength of Hübl's solution is shown by the following experiment: 25 grammes of iodine and 30 of mercuric chloride were dissolved each in 500 c.c. of 95 per cent. alcohol, and the solutions mixed. In five minutes' time the free iodine was 24.7 grammes, and the hydrochloric acid (calculated into iodine) 0.20 gramme per litre. After 85 days, the iodine had fallen to 10.82 grammes, and the acid risen to 14 grammes per litre. This is probably due to the water in the spirit, and not, as stated by Hübl, to the impurities present. It may be obviated to some extent by the use of absolute alcohol, but in the latter case, the small amounts of moisture taken up during the absorption process disturb the results more than when proper allowance is made for the diminished strength of the reagent.

To overcome this difficulty, the author saturates the iodine solution, on preparation, with hydrochloric acid. Twenty-five grammes of iodine are dissolved in 250 c.c. of (95 per cent.) alcohol, 25 grammes of mercuric chloride in 200 c.c. of alcohol added, then 25 grammes of hydrochloric acid, specific gravity 1.19, and the whole made up to 500 c.c. Such a liquid was found, after one hour, to contain 49.31; after 5 days, 49.18; after 20 days, 48.5; and after 64 days, 46.60 grammes of iodine per litre; while the free acid had only risen by an amount equal to 2.54 grammes of iodine. It will be observed that this solution is double the usual strength, but the results obtained agree exactly with those by the ordinary method. The explanation of this must be that the oxygen produced during the action of Hübl's solution is absorbed by the fat only in amount corresponding to the degree with which it still lacks halogen to render it completely saturated. Were this not the case, it would not be evident why the reaction between the iodine and the mercuric chloride should not continue as long as there are unsaturated fatty acids present. The above experiments, however, with varying weights of mercuric chloride, show that it does not so continue.

"Hübl's iodine number," therefore, is in reality the amount, expressed as iodine, of the chlorine, iodine, and oxygen taken up by the fat examined, together with the small amount of chlorine set free from the mercuric chloride by the natural decompo-

sition of the solution during the time of action; while the true iodine number should exclude this latter factor. It is, however, very small in amount, and there seems little reason to substitute for the ordinary process—beyond the improvement suggested above—any method based on the absorption of bromine. F. H. L.

Quantitative Estimation of Cellulose. Gerhard Lange. (*Zeit. angew. Chem.*, 1895, xix., pp. 561-563.)—In 1889 (*Zeit. phys. Chem.* xiv., 3, p. 283) the author published a method for estimating cellulose, in which the substance was heated with pure alkali and some water to about 150° C. in an oil-bath, the cellulose being subsequently collected, washed, dried, and weighed. Recent experiments have proved that by conducting the operation at the higher temperature of 180° C., a purer cellulose is obtained, containing only a very small amount of nitrogenous matter.

In the amended process a retort is no longer used, but a wide unglazed porcelain crucible, about 65 mm. high. From 5 to 10 grammes of the substance under examination are moistened with a little water in the crucible, and three times the weight of caustic potash (free from nitrate) added, together with about 20 c.c. of water. The crucible is then immersed in an oil-bath, so that the oil is at the same level as the contents of the crucible. The mass is continually stirred with the thermometer from the time frothing commences. As soon as the action has ceased, the crucible is covered with a lid having an opening for the thermometer, maintained at 175° to 180° C. for an hour, and then removed from the oil-bath.

When the temperature has fallen to about 80° C., 75 c.c. of hot water are added, and the mass allowed to cool, after which it is cautiously acidified with sulphuric acid, and washed into a large centrifugal tube. Caustic soda is next added to feebly alkaline reaction, and now nothing but the cellulose remains undissolved.

By energetic whirling in the centrifugal machine the cellulose separates completely and coagulates. The clear liquid is poured off, if necessary through a weighed filter, the cellulose broken up with a glass rod, hot water added, and the tube whirled again. The cellulose is then filtered off, washed with hot water, alcohol, and ether, dried and weighed. The amount of ash is also determined, and this deducted from the weight of the filter and the cellulose gives together the amount of the latter.

With a little practice the estimation can be brought to the drying stage in about two hours and a half. C. A. M.

The Determination of Tannin by Metallic Oxides. William H. Krug. (*Journ. Amer. Chem. Soc.*, xvii., 1895, pp. 811-814.)—The object of the author's investigation was to discover whether it were possible to substitute a metallic oxide for hide-powder, as used in the ordinary shaking method. The oxides tried were those of mercury, magnesium, zinc, and lead. In each case it was found that after shaking for some time combination was incomplete, and that a further period of standing was necessary to bring this about. This varied considerably with the different oxides. With magnesium and mercury the mixture was ready for filtering after being shaken for four hours and standing all night. With lead oxide two days' standing was required, while zinc oxide stood a week before the absorption of tannin was complete.

The extract used contained 42.35 per cent. soluble solids, and the solution used in the experiments contained 2 grammes of this extract per 100 c.c. Twenty-five c.c. of this dilute extract gave 0.0113 gramme of ash, containing 0.0006 gramme of magnesium oxide.

The tannin was determined by evaporating 25 c.c. of the filtrate, and deducting the dried residue from the weight of the solids in 25 c.c. of the original dilute extract. The ash determinations served to check the amount of oxide dissolved.

EXPERIMENTS WITH ZINC OXIDE.

Method.	Grammes ZnO used.	Diluted Extract used, c.c.	C.c. of Filtrate evaporated.	Residue, Grammes.	Per cent. Tannin in Extract.	Ash in Residue, Grammes.	ZnO in Ash, Grammes.
Shaken 4 hours. Stood over-night ...	2.0	75	25	0.1095	20.45	0.0125	0.0008
Shaken 4 hours. Stood over-night ...	2.0	75	25	0.1174	18.87	0.0147	0.0019
Shaken 4 hours. Stood a week ...	2.0	75	25	0.1049	21.37	0.0095	—
Shaken 4 hours. Stood a week ...	2.0	75	25	0.1029	21.97	0.0103	0.0030
Shaken 4 hours. Stood a week ...	2.0	75	25	0.1029	21.97	0.0010	0.0020

In the two first cases the filtrate showed a tannin reaction, and in the other three the whole of the tannin was not absorbed until after seven days. For this reason zinc oxide would be valueless where rapid work was required. The results obtained with it were lower than those with lead and mercury oxides, but agreed better with the magnesium oxide figures.

EXPERIMENTS WITH LEAD OXIDE.

Method.	Grammes PbO used.	Diluted Extract, c.c.	Filtrate evaporated, c.c.	Residue, Grammes.	Per cent. Tannin in Extract.	Ash in Residue, Grammes.	PbO in Ash, Grammes.
Shaken 3½ hours. Stood 2 days ...	4.0	75	25	0.0738	27.59	0.0111	0
Shaken 3½ hours. Stood 2 days ...	4.0	75	25	0.0754	27.27	0.0092	0
Shaken 3½ hours. Stood 2 days ...	4.0	75	25	0.0706	28.23	0.0105	0

There was no trace of tannin in the filtrates, but the objection to the use of lead oxide is the time required for complete combination.

EXPERIMENTS WITH MAGNESIUM OXIDE.

Method.	Grammes MgO used.	Diluted Extract, c.c.	Filtrate evapo- rated, c.c.	Residue, Grammes.	Per cent. Tannin in Extract.	Ash in Residue, Grammes.	MgO in Ash, Grammes.
Shaken 2½ hours. Stood over-night ...	2.0	75	25	0.1134	19.67	0.0209	0.0055
Shaken 2½ hours. Stood over-night ...	2.0	75	25	0.1028	21.79	0.0195	0.0042
Shaken 2½ hours. Stood over-night ...	2.0	75	25	0.1230	17.75	0.0232	0.0052
Shaken 2½ hours. Stood over-night ...	2.0	75	25	0.1246	17.43	0.0247	0.0058
Stood 24 hours, with occasional shaking...	2.0	75	50	0.2063	21.72	0.0252	0.0074
Stood 24 hours, with occasional shaking...	2.0	75	50	0.2061	21.74	0.0244	0.0076
Stood 24 hours, with occasional shaking...	2.0	75	25	0.1157	19.21	0.0213	0.0044
Stood 24 hours, with occasional shaking...	2.0	75	25	0.1114	20.07	0.0205	0.0038
Stood 48 hours, with occasional shaking...	2.0	75	50	0.1941	22.94	0.0326	0.0064
Stood 48 hours, with occasional shaking...	2.0	75	50	0.1959	22.76	0.0339	0.0070
Shaken 3½ hours. Stood over-night ...	2.0	75	50	0.2165	20.70	0.0349	0.0102
Shaken 3½ hours. Stood over-night ...	2.0	75	50	0.2191	20.44	0.0355	0.0111
Shaken 3½ hours. Stood over-night ...	2.0	75	50	0.2179	20.56	0.0359	0.0115

The amount of oxide dissolved was considerable, and lowered the tannin correspondingly. Since this error was not constant, no correction for it appeared possible.

EXPERIMENTS WITH MERCURIC OXIDE (YELLOW).

Method.	Grammes HgO used.	Diluted Extract, c.c.	Filtrate evapo- rated, c.c.	Residue, Grammes.	Per cent. Tannin in Extract.	Ash in Residue, Grammes.	HgO in Ash, Grammes.
Stood 48 hours, with occasional shaking...	4.0	75	25	0.0833	25.69	0.0070	0
Stood 48 hours, with occasional shaking...	4.0	75	25	0.0829	25.77	0.0077	0
Stood 48 hours, with occasional shaking...	4.0	75	25	0.0829	25.77	0.0071	0
Shaken 1½ hours. Stood over-night ...	4.0	75	25	0.0842	25.51	—	—
Shaken 2 hours. Stood over-night ...	4.0	75	25	0.0867	25.01	—	—

EXPERIMENTS WITH MERCURIC OXIDE (YELLOW) *continued*—

Method.	Grammes HgO used.	Diluted Extract, c.c.	Filtrate evapo- rated, c.c.	Residue, Grammes.	Per cent. Tannin in Extract.	Ash in Residue, Grammes.	HgO in Ash, Grammes.
Shaken 3½ hours. Stood over-night	4.0	75	25	0.0863	25.09	0.0081	0
Shaken 3½ hours. Stood over-night	4.0	75	25	0.0860	25.15	0.0080	0
Shaken 4 hours. Stood over-night	4.0	75	25	0.0839	25.57	0.0084	0
Shaken 4 hours. Stood over-night	4.0	75	25	0.0849	25.37	0.0065	0
Shaken 4 hours. Stood over-night	4.0	75	20	0.0638	26.41	0.0065	0
Shaken 4 hours. Stood over-night	4.0	75	20	0.0692	25.05	0.0103	0
Shaken 4 hours. Stood over-night	4.0	75	20	0.0682	25.29	0.0094	
Shaken 4 hours. Stood over-night	4.0	75	25	0.0863	25.09	0.0083	0
Shaken 4 hours. Stood over-night	4.0	75	25	0.0849	25.37	0.0097	0
Shaken 4 hours. Stood over-night	4.0	75	25	0.0842	25.51	0.0084	0

The foregoing results show that even under variable conditions constant results may be obtained. The end of the reaction may be recognised by the whole mass partially gelatinizing and becoming a dirty brown. The amount of ash held back is fairly constant, and may be neglected. The time required is greater than that with hide-powder, but the uniform results obtained compensate for this.

C. A. M.

The Analysis of Lead: Metal and Ores. H. Nissenson and B. Neumann. (*Chem. Zeit.*, 1895, xix., 1141-1143.)—*Soft Lead.*—For a proper valuation of this material, more especially when it is to be used for the manufacture of pigments, it is necessary to determine the whole of the impurities present. The following scheme includes silver, copper, bismuth, cadmium, zinc, iron, nickel and cobalt, tin and antimony (arsenic and manganese being very seldom detected). 200 grammes of the metal in small pieces are put into a 2-litre flask with 1275 c.c. of water, 325 c.c. of nitric acid (sp. gr. 1.4) are added, and the whole warmed on the sand-bath till dissolved. The liquid is cooled and the lead precipitated with 62 c.c. of strong sulphuric acid. It is not necessary to allow the precipitate to settle for a long time (*cf.* Fresenius), as modern soft lead only contains antimony to the third place of decimals of a per cent., nor need hydrochloric acid be added to throw down the silver. After cooling the liquid is made up to 2 litres, well shaken, and when again clear 1750 c.c. are filtered off and evaporated to dryness in a porcelain basin. The residue is treated with a little water, filtered and washed. The insoluble portion is digested in an Erlenmeyer flask with caustic

soda and 25 c.c. of cold saturated sodium sulphide solution to dissolve the antimony, and the liquid (A) is filtered off to be subsequently added to the bulk of the antimony. The main filtrate, which if not acid is made so with a few drops of hydrochloric acid, is treated with sulphuretted hydrogen till the liquid becomes perfectly clear, and is separated into a precipitate (C) and a filtrate (D). To C 25 c.c. of sodium sulphide are added, which dissolves the antimony, tin, and arsenic. In the absence of arsenic, the dissolved portion is mixed with A, made up to 200 c.c. and electrolyzed at the boil in a platinum basin for half an hour with a current of 1.2 ampères and 2 volts ($N.D_{100} = 1.2$ ampère), to remove the *antimony*. The *tin* is estimated either by destroying the sodium sulphide with 25 grammes of solid ammonium sulphate and electrolyzing for twenty minutes, or the solution may be acidified with dilute sulphuric acid and the tin sulphide ignited gently, moistened with nitric acid and weighed as oxide. In the presence of arsenic the solution containing the three sulphides must be acidified with weak sulphuric acid, the *arsenic* removed from the precipitate by cold ammonium carbonate (to be estimated either as sulphide or as ammonio-magnesium arseniate) and the antimony and tin dissolved in sodium sulphide and separated as above.

The insoluble portion of C is boiled with aqua regia, the silver chloride removed, the liquid evaporated to dryness with a little sulphuric acid to get rid of the lead, the residue taken up in water, and ammonia and ammonium carbonate added. The precipitate of *bismuth* is filtered off, dissolved in nitric acid, evaporated in a tared basin and weighed as Bi_2O_3 . In the solution is dissolved 1 gramme of potassium cyanide, and the *cadmium* thrown down with a few drops of sodium sulphide, to be treated like the bismuth precipitate. The potassium cyanide is then destroyed by boiling with sulphuric acid, the solution diluted to 200 c.c., and the *copper* deposited on a small and light electrode by a current of 1.1.5 ampère and 2.5.3 volts passing for one to two hours. The acid liquid may also be treated with 0.5 gramme of solid sodium thiosulphate, and the copper sulphide weighed as oxide.

The liquid D is boiled till it becomes cloudy, oxidized with bromine-water, and caustic soda added. The precipitation is repeated a second time to ensure all the zinc remaining dissolved. The precipitate is dissolved in dilute sulphuric acid, and the *iron* thrown down twice with ammonia, and determined volumetrically with permanganate. The filtrate is made alkaline with ammonia, 25 grammes of ammonium sulphate added, and the *cobalt* and *nickel* recovered by an electric current of 1.5 ampère, 2.5.3 volts for half an hour. The alkaline *zinc* solution is acidified, excess of ammonia added, made up to 500 c.c., and titrated by Schaffner's method with sodium sulphide, using lead-paper as an indicator.

The *silver* is determined in a special portion of the metal by cupellation, and the lead is estimated by difference. All the above results are calculated on 179.12 grammes of lead, as the original precipitate of sulphate occupies 46 c.c. (1954 : 200 :: 1750 : 179.12).

Hard or Antimonial Lead.—Here it is only necessary, as a rule, to estimate the antimony and the copper. Fresenius' method for the former (*Quant. Anal.*, ii., 483) is objectionable, owing to the repeated filtrations required; and the fusion processes are tedious. 2.5 grammes of the alloy, 10 grammes of tartaric acid, 15 c.c. of water,

and 4 c.c. of 1.4 nitric acid, are put into a 250 c.c. flask and dissolved. 4 c.c. of strong sulphuric acid are added, and when cold made up to the mark. The lead sulphate is perfectly free from antimony. 50 c.c. of the filtrate are made strongly alkaline with caustic soda, 50 c.c. of sodium sulphide added, boiled, filtered immediately, and the liquid electrolyzed hot for an hour to yield the antimony. The copper may be obtained from the undissolved portion either by electrolysis or determined by colorimetry.

Commercial leads are dissolved as above, from 5-10 grammes of tartaric acid being added to the solution of 10-50 grammes of the metal. The lead is precipitated with 3 c.c. of strong sulphuric acid for each 10 grammes of lead, and if only a portion of the liquid is taken for analysis, the same allowance of 2.15 c.c. (*sic cf. ante*) per 10 grammes of metal is made. The solution is concentrated, treated with caustic soda and sodium sulphide, and the dissolved antimony, arsenic and tin treated as above. The residue is dissolved in aqua regia, evaporated to dryness, dissolved in hydrochloric acid, filtered from the silver and treated with sulphuretted hydrogen. Both precipitate and filtrate are treated as before. When it is required only to know the amount of silver, copper and antimony present, the following method suffices: The silver is determined by cupellation, the antimony by electrolysis, and the residue insoluble in sodium sulphide is dissolved in aqua regia, supersaturated with ammonia and the copper determined colorimetrically, care being taken that the liquid for examination contains not more than 1 per cent. of copper.

Galena.—It is often necessary only to determine the lead, silver, arsenic and zinc in this material. The silver is estimated as before, but the lead is determined directly (especially when antimony and copper are present). 0.5 gramme is dissolved in 30 c.c. of 1.4 nitric acid, diluted, and electrolyzed hot in a platinum basin with a current of 1.1.5 ampère, 2.5 volts ($N.D_{100} = 1$ ampère), the basin itself being the anode. The peroxide is dried at 180° C. and weighed. For the arsenic, 1 gramme is dissolved, evaporated with sulphuric acid, diluted and filtered. To the filtrate, 5 grammes of tartaric acid, 30 c.c. of ammonia, 15 c.c. of magnesia mixture are added. The precipitate is allowed to settle, filtered off, dissolved in nitric acid, run into a porcelain basin, evaporated and ignited (phosphoric acid is never present). For the zinc, 2 grammes are boiled with hydrochloric acid till all the sulphuretted hydrogen is driven off, 5 c.c. of nitric and 8 c.c. of dilute sulphuric acid added, and the whole evaporated to half the volume. The liquid is diluted, 50 c.c. of ammonia added, boiled, and when cold made up to 500 c.c. Half the liquid is taken and titrated with sodium sulphide as above.

In the case of sulphides of lead and copper containing silver, etc., the two metals cannot be separated directly by electrolysis; the lead is thrown down as above, the peroxide remaining in the acid liquid for a few moments after the current has ceased, to free it from traces of copper; then the liquid is evaporated with sulphuric acid to dryness, dissolved in water, and the copper precipitated with thio-sulphate.

F. H. L.

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INDEX TO VOLUME XXI.

- ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS, 15, 44, 64, 102, 133, 158, 191, 209, 231, 255, 285, 332
- Acetanilide, Detection of, 291
- , Qualitative examination of, 138
- , Phenacetin, etc., Scheme for the identification of, 69
- Acetnaphthylamine, α , Action of nascent Bromine on, 238
- , β , Action of nascent Bromine on, 238
- Acetylene, Toxicity of, 66
- Acetyl-para-amido-phenetol, 70
- Acid, Boric, Estimation of, in Milk, 64
- , Carbonic, apparatus, New form of, 62
- , Fatty, New unsaturated, 298
- , Gallic, 296
- , Hydrochloric, Determination of, in the Gastric Juice, 74
- , Hydrocyanic, Detection of, in cases of Poisoning, 160
- , Hydrofluoric, Examination of, 243
- , ———, Note on the preparation of pure, 87
- , Isanic, 298
- , Molybdic, as a test for Alcohol, 163
- , Palmitic, 317
- , Phosphoric, Estimation of, Critical examination of the processes for the, 110
- , ———, ———, in Medicated Wines, 292
- , ———, ——— available, in Basic Slag, 81
- , ———, Gravimetric method of estimating, as Ammonium phospho-molybdate, 219
- , Picric, New reaction for, 332
- , Stearic, Determination of, in Fats, 316
- , Sulphuric, Determination of, or of Barium, 302
- , ———, Quantitative estimation of Arsenic in crude concentrated, 166
- , ———, and Potassium permanganate, Method for the standardization of, 245
- , Tartaric, Estimation of free, in Wines, 234
- , ———, the total, in crude Tartar and Wine Lees, 75
- , Titanic, and Iron, Estimation of, in ores, 77
- , Uric, Estimation of, in Guano, 332
- , ———, Method of estimating, 104
- , ———, Volumetric determination of, 212
- Acids, Arsenic and Phosphoric, Volumetric process for the determination of, 248
- , or Phosphoric, Estimation of Uranium in ores which contain, 26
- , Molybdic and Vanadic, Reactions of, with Thiocyanates, 273
- , Phosphoric and Arsenic, Volumetric process for the determination of, 248

- Acids, Phosphoric or Arsenic, Estimation of Uranium in ores which contain, 26**
 ———, Selenious and Selenic, Iodometric estimation of, 221
 ———, Vanadic and Molybdic, Reactions of, with Thiocyanates, 273
 ———, Volatile Fatty, Leffmann-Beam process for the estimation of, 259
 ——— in Wine, Estimation of the Volatile, 158
 ——— of the Fatty Series, Distillation of the lower, in presence of water, 239
Acorn Oil, 74
Agricultural Analysis, Principles and Practice of, Review, 249
Aikman, C. M., Milk, its Nature and Composition, Review, 55
 ———, and Wright, R. P., *Translators*, Book of the Dairy, *Review*, 306
Air-bath, Asbestos, 28
Albumin, Determination of, in Urine, 214
Albumoses, Zinc sulphate as a precipitant for, 16
Alcohol, Molybdic Acid as a test for, 163
 ——— and Extract in Wine, Estimation of, by the Refractometer, 133
 ——— as a source of error in the titration of Alkaloids, 293
 ——— in Wine, Estimation of, by the Ebullioscope and Distillation, 210
Alcoholic Indicator solutions, Decomposition of, 247
Alcohols, Estimation of Ethers in, 294
Aldehyde, Formic, Note on Hehner's test for, 157
 ———, ——— the estimation of, 148
Aldehydes, Differentiation of, by means of Phenols, and vice versa, 295
Alimentos y Bebidas, Investigacion de Sus. Alteraciones y Falsificaciones, Review, 306
Alkaloids, Alcohol as a source of error in the titration of, 293
 ———, Estimation of, by titration with Iodine solution, 191
 ——— of the Kola-nut, 292
Allen, A. H., Commercial Organic Analysis, 2nd Edn., Review, 279
 ———, Composition and analysis of commercial Cream of Tartar, 174
 ———, Estimation of Lead in Water, 13
 ———, Food adulteration, 116
 ———, Ginger, 313
 ———, Note on the analysis of Cream of Tartar, 209
 ———, ——— concentration of Condensed Milk, 281
 ———, ——— preparation of pure Hydrofluoric Acid, 87
 ———, ——— titration of Quinine, 85
 ———, White-Wine Vinegar, 253
Almond Oil, 328
Alumina and Iron oxide, Determination of, in Phosphate rock by the Ammonium
 ——— Acetate method, 272
 ———, New method of estimating, in Phosphate rock, 273
Aluminium and its Alloys, Analysis of, 83, 270
Amines, Determination of Nitrogen in the compounds of, with metallic Chlorides, 71
Ammonia, Separation of Trimethylamine from, 263
Ammonium citrate, Action of, on Basic Slag, 166
 ——— Ferrous sulphate, Inversion of Sugar by, 136
 ——— Molybdate solution, Modified, 220
Amsel, H., Resinated Metallic Oxides, 261
Aniline and Ortho- and Para-Toluidines, Estimation of moisture in, 215
 ——— Toluidine, Estimation of, in presence of small quantities of each other, 215
Animal and Vegetable Oils, Detection of, in Mineral Oils, 105
 ——— Oil, 243
Antipyrine, 70
Antusch, A. C., and Van Ketel, B.A., Examination of Linseed-cake Oil, 299
APPARATUS. ABSTRACTS, 27, 84, 248, 277, 304

APPOINTMENT :

- Sutton, W. L., as Public Analyst for Norwich, 28
 Aqua Regia, Toxicological detection of, 21
 Araeometer Pipette, 249
 Arsenic, Quantitative estimation of, in crude concentrated Sulphuric Acid, 166
 ——— and Phosphoric Acids, Volumetric process for the determination of, 248
 ——— or Phosphoric Acids, Estimation of Uranium in ores which contain, 26
 Arsenical Sulphuretted Hydrogen, Purification of by Iodine, 236
 Asbestos Air-bath, 28
 Asboth. *See* Von Asboth
 Aschman, C., Examination of Butter, 287
 Asparagine, New reaction for, 332
 Asphaltum, Technical analysis of, 161, 211
 Asses' Milk, Composition of, 88
 Atkinson, F., and Smith, E. F., Separation of Iron from Beryllium, 23
 Auchy, G., Sources of error in Volhard's and similar methods of determining Manganese in Steel, 334

 Bacillus coli communis, 120
 ——— typhosus, 120
 Bailhache, G., and Rivière, G., Kjeldahl's method and its modifications, 267
 Balance, Bunge's improved analytical, 248
 ———, Westphal, Note on the use of the, 156
 Barbet and Jandrier, Mm., Differentiation of Aldehydes by means of Phenols, and vice versa, 295
 ———, Estimation of Ethers in Alcohols, 294
 Barium, Determination of Sulphuric Acid or of, 302
 Basic Slag, Action of Ammonium citrate on, 166
 ———, Estimation of available Phosphoric Acid in, 81
 Bath, Air, Asbestos, 28
 ———, Water, Koch's Sterilizing Cylinder as a, 27
 Bau, A., Koch's Sterilizing Cylinder as a Water-bath, 27
 Bauer, E., Estimation of Potassium, 217
 ———, F., and Hilger, A., Examination of Peppers, 257
 Baumert, G., Quantitative estimation of Cellulose in Foods, 256
 Beam. *See* Leffmann-Beam
 Beckmann, E., Contributions to the analysis of Honey, 287
 Beef fat, Note on the microscopic detection of, in Lard, 254
 Beer, Detection of Fluorine in, 15
 Bees Wax, 192
 Belar, A., Detection of foreign Colouring matters in Red Wines, 289
 Benzene, Detection of, in Petroleum Spirit, and vice versa, 69
 ———, Volumetric estimation of Thiophen in, 297
 Benzoates, Alkaline, New method of analysing, 103
 Bergamot, Oil of, Examination of, 137
 Beryllium, Separation of Iron from, 23
 Bishop, W., Determination of the degree of oxidation of Oils, 105
 Bismuth sulphide, Solubility of, in Sodium sulphide, 335
 Biuret reaction, 103
 Blasdale, W. C., Some Californian Oils, 73
 Blount, B., Determination of Oxygen in commercial Copper, 57
 Blue, Prussian, Commercial, 225
 Blunt, T. P., Note on Ginger, 309
 Boilers, Silica a cause of Scale in, 139

- Bömer, A., Zinc sulphate as a precipitant for Albumoses, 16
 ———, and König, J., Composition of Meat Extracts, 17
- BOOKS, REVIEWS OF:
- Aikman, C. M., *Milk, its Nature and Composition*, 55
 - , and Wright, R. P., *Translators, Book of the Dairy*, 306
 - Allen, A. H., *Commercial Organic Analysis*, 2nd Edn., 279
 - Chicote, Dr. C., *Alimentos y Bebidas, Investigacion de Sus, Alteraciones y Falsificaciones*, 306
 - Clowes, F., *Practical Chemistry and Qualitative Analysis*, 6th Edn., 28
 - , and Coleman, J. B., *Quantitative Chemical Analysis*, 3rd Edn., 28
 - Coleman, J. B., and Clowes, F., *Quantitative Chemical Analysis*, 3rd Edn., 28
 - Fleischmann, Prof., *Book of the Dairy*, 306
 - Hansen, E. C., *Practical Studies in Fermentation*, 112
 - Holloway, G. T., and Redwood, B., *Petroleum*, 167
 - Miller, Dr. A. K., *Translator, Practical Studies in Fermentation*, 112
 - Redwood, B., and Holloway, G. T., *Petroleum*, 167
 - Sadtler, S. P., *Handbook of Industrial Organic Chemistry*, 2nd Edn., 279
 - Sanford, P. G., *Nitro-Explosives*, 277
 - Sutton, F., *Systematic Handbook of Volumetric Analysis*, 7th Edn., 308
 - Wiley, H. W., *Principles and Practice of Agricultural Analysis*, 249
 - Wise, W. L., *Gleanings from the Patent Laws of all Countries*, 28
 - Wright, R. P., and Aikman, C. M., *Translators, Book of the Dairy*, 306
- Borax, Detection of, in Butter, 286
- Boric Acid, Estimation of, in Milk, 64
- Borutriger, A., Examination of Oil of Bergamot, 137
- Boseley, L. K., and Richmond, H. D., Further notes on the detection of Formalin, 92
- Brand, J., Detection of Fluorine in Beer, 15
- Bread, Note on Ammunition, 255
- and Meal, Detection of Ergot in, 15
- Brenzinger, K., Estimation of Para-Sulphanilic Acid, 214
- Bridge v. Howard, Milk Certificate case, 305
- Brociner, L., Toxicity of Acetylene, 66
- Bromine, Action of nascent, on Naphthols and Naphthylamine, 237
- and Chlorine, Quantitative separation of, 79
- Iodine, New reagent for, 68
- Brown, J. C., Ginger, 310
- Brüll, J., and Ulzer, F., Estimation of Manganese in pig Iron, 139
- Brunswick Green, Analysis of, 230
- Budden, E. R., Note on a convenient form of Polarimeter for examining Essential Oils, 14
- , and Hardy, H., Note on the estimation of minute quantities of Metals in Liquids, 12
- Bugarski, S., Quantitative separation of Chlorine and Bromine, 79
- Bunge's improved analytical Balance, 248
- Butter, 258, 321, 329
- , Cocoa, 328
- , Iodine number and refractive index of, 231
- , Composition of, 89
- , Detection of Borax in, 286
- , Estimation of Water in, 242
- , Examination of, 287
- and Lard analyses, Use of the Calorimeter in, 107
- and Milk certificates, 113

- Cabannes, M., Adulteration of Cantharides, 290
 Cadmium chloride, Inversion of Sugar by, 137
 Caffeine, Estimation of, 232
 ———, ———, by means of Wagner's reagent, 193
 ———, ———, in Tea, 232
 Calcium oxalate, Precipitation of, 244
 Californian Oils, Some, 73
 Cameline seed, 299
 Cameron, Sir C. A., Note on ammunition Bread, 255
 Campbell, E. D., and Hart, E. B., Quantitative determination of Hydrogen, 222
 ———, G. F., and Osborne, T. B., Chemical nature of Diastase, II., 257
 ———. See Hayes-Campbell
 Camphor, 296
 Cantharides, Adulteration of, 290
 Caraways, Note on "drawn" or exhausted, 207
 Carbon and Sulphur, Estimation of, in Zinc, 76
 Carbonic Acid apparatus, New form of, 62
 Carbylamine reaction, 69
 Carles, P., Analysis of the Kola nut, 265
 ———, ———, Alkaloids of the Kola nut, 292
 Carnauba Wax, 193
 Carnot, A., Analysis of a mixture of Chlorides, Chlorates, and Perchlorates, 220
 ———, Volumetric determination of mixtures of Chlorides, Hypochlorites, and Chlorates, 221
 ———, ———, Separation of Cobalt and Nickel, 52
 ——— method, Estimation of Potash by the, 216
 Carteighe, M., Public Analysts, 41
 Caspari, C., Alcohol as a source of error in the titration of Alkaloids, 293
 Cassal, ———, Food adulteration, 115
 ———, Public Analysts and Somerset House, 41
 Causse, ———, Estimation of Glucose, 238
 Cellulose, Quantitative Estimation of, in Foods, 256
 Cerhez, S., Asbestos Air-bath, 28
 Certificates, Milk, *Bridge v. Howard*, 305
 ———, ———, *Fortune v. Hanson*, 53
 ———, ———, and Butter, 113
 Chambers, A. D., and Morse, H. N., Method for the standardization of Potassium permanganate and Sulphuric Acid, 245
 Chapman, A. C., Diastatic power of Malt, 127
 Cheese, Canadian, Lead in a sample of, 208
 ———, Extraction of the fat of, for the purpose of examination, 44
 Chicote, Dr. C., Alimentos y Bedidas, Investigacion de Sus Alteraciones y Falsificaciones, *Review*, 306
 Chlorates, Colour reaction for, 51
 ———, Chlorides, and Hypochlorites, Volumetric determination of mixtures of, 221
 ———, ———, Perchlorates, Analysis of a mixture of, 220
 Chlorides, Chlorates, and Perchlorates, Analysis of a mixture of, 220
 ———, Hypochlorites, and Chlorates, Volumetric determination of mixtures of, 221
 Chlorine and Bromine, Quantitative separation of, 79
 ——— Sulphur, Estimation of, by means of Sodium Peroxide, 49
 Chocolate, Estimation of Sugar in, 256
 Christensen, A., Volumetric process for the determination of Phosphoric and Arsenic Acids, 248
 Cinchona, Extract of, Valuation of, 47

- Cinnabar, Electrolytic method for the determination of Mercury in, 165
 Cloedt. *See* Von Cloedt
 Clotted Cream, Composition of, 89
 Clowes, F., Practical Chemistry and Qualitative Analysis, 6th Edn., *Review*, 28
 ———, and Coleman, J. B., Quantitative Chemical Analysis, 3rd Edn., *Review*, 28
 Coals, Determination of the heating effects of, 21
 Cobalt and Nickel, Separation of, 52
 Cochius, F., Improved Gas-measuring vessel, 84
 Cocoa Butter, 328
 ———, Iodine number and refractive index of, 231
 Coconut Oil, 107
 Coleman, J. B., and Clowes, F., Quantitative Chemical Analysis, 3rd Edn., *Review*, 28
 Colophony, 261
 Coloration of Wine, Artificial, 45
 Colouring matter of natural Waters, 209
 ——— matters, Detection of foreign, in Red Wines, 289
 Colours, Oil-, Linseed Oil and Varnish, Examination of, 46
 Colza Oil, 107, 243
 Commercial Organic Analysis, 2nd Edn., *Review*, 279
 Condensed Milk, Note on the concentration of, 281
 Condenser, Liebig, 84
 Cone, E. F., Estimation of Pyrrhotite in Pyrites ores, 223
 Copper, Amount of, absorbed by Vegetables from a Coppery soil, 290
 —, in Vegetable produce, 235
 — Determination of Oxygen in commercial, 57
 — Estimation of, by the cyanide process, 167
 — Volumetric estimation of, 110
 — and its ores, Analysis of, 51
 — in Wine, 102
 Coremons and Courlay, Mm., Chemical detection of Horseflesh, 231
 Cornette, P., Estimation of Resin in Fatty Oils, 260
 CORRESPONDENCE, 140, 168, 224
 Coste, J. H., and Parry, E. J., Commercial Prussian Blue, 225
 Cotton Oil, 258, 259
 ———, "Stearin," 328
 Cotton-seed Oil, 106
 Courlay and Coremons, Mm., Chemical detection of Horseflesh, 231
 Cows' Milk, Variation in size of the fat-globules in, 44
 Cox, F. H., Ginger, 316
 Craver, H., Noyes, W., and McTaggart, J., Determination of the heating effects of
 Coals, 21
 Cream, Clotted, Composition of, 89
 ———, Composition of, 88
 ———, Milk, and skimmed Milk, Relative composition of, 283
 ——— of Tartar, Composition and analysis of commercial, 174
 ———, Note on the analysis of, 209
 Cribb, C. H., New form of Carbonic Acid apparatus, 62
 Crismer, L., Resazurine as an Indicator, 245
 ———, and Motteu, J., Saturation temperatures and Critical temperatures.
 Application to general analysis, 241
 Critical temperatures and Saturation temperatures. Application to general analysis,
 241
 Crolas, —, Urine reducing Fehling solution, 334
 Cubebs, Notes on, 236

- Cuprous Oxide as a reagent for Nitrites, 246
- Cushman, A. S., and Hayes-Campbell, J., Volumetric determination of Lead, 48
- Dairy, Book of the, *Review*, 306
- Dam. See Von Dam
- Darnel, Testing for, in Flour, 231
- De Koninck, L. L., Acidimetric determination of Zinc, 222
- _____, Influence of Magnesia mixture on Glass, 166
- _____, Standardizing volumetric apparatus, 249
- De la Source, L. M., Determination of the dry extract of Wine, 66
- _____, Estimation of free Tartaric Acid in Wines, 234
- _____, the Volatile Acids in Wine, 158
- De Rochefontaine, D'H., Estimation of the total Tartaric Acid in crude Tartar and Wine Lees, 75
- De Schweinitz, E., and Emery, J. A., Use of the Calorimeter in Butter and Lard analyses, 107
- Delepine, M., Method for the separation of Methylamines, 297
- Denamur, V., Commercial analysis of Glucose, 194
- Denigès, C., Detection of Formaldehyde in Milk, 285
- _____, Rapid detection of Tin, 23
- _____, Estimation of Copper by cyanide process, 167
- _____, General method for the estimation of Mercury, 303
- _____, Nessler's reaction as a test for Mercury or Iodides, 77
- _____, Volumetric determination of Mercury by Potassium iodide, 247
- _____, Uric Acid, 212
- _____, estimation of Thiophen in Benzene, 297
- _____, M. G., Colour reaction for Chlorates, 52
- _____, Three new reagents for the detection of Nitrites, 51
- Deposits formed in Water-pipes, Note on the composition of two, 169
- Deros, A., Testing for Darnel in Flour, 231
- Diastase, Chemical nature of, 267
- Dibdin, W. J., Microscopical examination of Water, 2
- _____, and Grimwood, R., Analysis of Mortar, 197
- Dieterich, K., Microscopy of Honey, 255
- Digitalein, 46
- Digitalin, 46
- _____, Reactions of, 46
- Digitonin, 46
- Digitoxin, 46
- Disinfectants and Soaps, Estimation of Phenol in, 301
- Dobriner, P., and Schranz, W., Estimation of Aniline and Toluidine in presence of small quantities of each other, 215
- _____, moisture in Aniline and in Ortho- and Para-Toluidines, 215
- Dougall, J. D., Standard Potassium dichromate solution, 82
- "Driers," Composition of, 300
- Drug and Food legislation, 36
- DRUGS AND FOOD ANALYSIS. ABSTRACTS. See FOOD AND DRUGS ANALYSIS. ABSTRACTS.
- Drumel, L., Composition of Rabbits' fat, 135
- Dulcin, New test for, 164
- Dupré, A., Examination of Water, 224
- Dyer, B., and Gilbard, J. F. H., Note on "drawn" or exhausted Caraways, 207
- Dynamite, Estimation of Nitro-glycerin in, 241

- Earthnut Oil, 106, 258, 328
 ———, Colour reaction for, 258
 Eberhardt, E. G., Examination of powdered Gamboge, 266
 Edinger, A., Estimation of Sulphur and Chlorine by means of Sodium peroxide, 49
 Editorial, 1
 Edmunds, J., Determination of Sulphuric Acid or of Barium, 302
 Egg, Oil of the, 161
 ———-yolk, Detection of, in Pastry, etc., 233
 Elion, H., Notes on the gravimetric estimation of Sugar by Fehling's solution, 334
 Elliott, T. H., Relations of Public Analysts to Government Boards, 40
 Ellms, J. W., and Richards, E., Colouring matter of natural Waters, 209
 Ellram, W., Reactions of Vanadic and Molybdic Acids with Thiocyanates, 273
 Emery, J. A., and De Schweinitz, E., Use of the Calorimeter in Butter and Lard analyses, 107
 Enamel, Pottery, Determination of Gold in, 140
 Engelen. *See* Van Engelen
 Ergot, Detection of, in Meal and Bread, 15
 Essential Oil of Mustard, Estimation of the, in Feeding Stuffs, 233
 ——— Oils, Note on a convenient form of Polarimeter for examining, 14
 Ethers, Estimation of, in Alcohols, 294
 Ethylene, Estimation of, in mixed Gases, 263
 Euler, H., and Friedheim, C., Estimation of Molybdenum, and the volumetric separation of Molybdenum and Vanadium, 25
 Ewell, E. E., and Wiley, H. W., Determination of Lactose in Milks, by double dilution and polarization, 182
 Exalgin, 70, 291
 Explosives, Nitro-, *Review*, 277
 Extract of Cinchona, Valuation of, 47

 Fabre, C., Estimation of Potash, 245
 Fairbanks, C., and Gooch, F. A., Estimation of the Halogens in the mixtures of their Silver salts, 24
 Fat, Horse, and Horse-flesh, 231
 ———, Human, Composition of, 171
 ———, Rabbits', Composition of, 135
 Fats, Determination of Stearic Acid in, 316
 ———, Measurement of rancidity of, other than Butter, 298
 ——— and Oils, Bromine heat-value of, 107
 ———, Electrical conductivity of, 257
 Fatty Acid, New unsaturated, 298
 ——— Acids, Volatile, Leffmann-Beam process for the estimation of, 259
 ——— Oils, Estimation of Resin in, 260
 ——— series, Distillation of the lower Acids of the, in presence of water, 239
 Feeding Stuffs, Estimation of the Essential Oil of Mustard in, 233
 Fehling solution, Urine reducing, 334
 Fehling's solution, Notes on the gravimetric estimation of Sugar by, 334
 Fermentation, Practical Studies in, *Review*, 112
 Ferro-Manganese, Inaccuracies in the estimation of Manganese in, 196
 Ferrous Ammonium sulphate, Inversion of Sugar by, 136
 ——— bromide, Inversion of Sugar by, 136
 ——— chloride, Inversion of Sugar by, 136
 ——— sulphate, Inversion of Sugar by, 136
 Fertilizers, Official methods for the analysis of, issued by the German Manure Manufacturers' Association, 99, 128, 151, 186

- Filsinger, F., Detection of Hydrocyanic Acid in cases of Poisoning, 160
 Fish Oil, Japanese, 243
 Fleck, H., Separation of Trimethylamine from Ammonia, 263
 Fleischmann, Prof., —, Book of the Dairy, *Review*, 306
 Flour, Testing for Darnel in, 231
 Flugge, Prof., —, Sterilization of Milk, 102
 Fluorine, Detection of, in Beer, 15
 Foerster, O., Action of Ammonium citrate on Basic Slag, 166
 Food, Rapid detection of Zinc in articles of, 290
 ——— adulteration, 115
 FOOD ANALYSIS. ABSTRACTS, 15, 44, 64, 102, 133, 158, 209, 231, 255
 Food and Drug legislation, 36
 FOOD AND DRUGS ANALYSIS. ABSTRACTS, 285
 Foods, Quantitative estimation of Cellulose in, 256
 Formaldehyde, Detection of, in Milk, 285
 ———, Reactions of, 102
 Formalin, Detection of, 94, 168
 ———, ———, Further notes on the, 92
 Formic Aldehyde, Note on Hehner's test for, 157
 ———, ——— the estimation of, 148
 ———, Tests for, 98
 Fortune v. Hanson, Milk Certificate case, 53
 Franke, E., Estimation of Nitrogen in Guano, 164
 Fresenius, C., Notes on Lard, etc., 159
 ———, H., and Makin, C. J. S., Estimation of Phenol in Soaps and Disinfectants, 301
 ———, R., and Hintz, E., Estimation of Uranium in ores which contain Phosphoric or Arsenic Acids, 26
 Friederichs and Greiner, Mm., Araeometer Pipette, 249
 Friedheim, C., and Euler, H., Estimation of Molybdenum, and the volumetric separation of Molybdenum and Vanadium, 25
 Fritzsche, P., Estimation of Ethylene in mixed Gases, 263
 Froidevaux, J., Potassium chromate as a Milk preservative, 285
 Frühling, R., Horse-flesh and Horse-fat, 231
 Fucks, F., and Schiff, F., Yellow tint of Zinc-white, 139
 Funk, R., Estimation of Sulphur and Carbon in Zinc, 76
 Fusel Oil, Estimation of, in rectified Spirit, 213

 Gallic Acid, 296
 Gamboge, Examination of powdered, 266
 Gas-measuring vessel, Improved, 84
 Gases, Estimation of Ethylene in mixed, 263
 Gastric Juice, Determination of Hydrochloric Acid in the, 74
 Gelatin, Estimation of, in Meat Extracts and commercial Peptones, 19
 Georges, M., Estimation of Caffeine, 232
 Gerlach, M., and Passon, M., Estimation of available Phosphoric Acid in Basic Slag, 81
 Gilbard, J. F. H., and Dyer, B., Note on "drawn" or exhausted Caraways, 207
 Gill, A. H., and Richardson, H. A., Determination of Nitrites in potable Water, 137
 Ginger, Note on, 309
 Gladding, T. S., Determination of Iron oxide and Alumina in Phosphate rock by the Ammonium acetate method, 272
 ———, Gravimetric method of estimating Phosphoric Acid as Ammonium phospho-molybdate, 219

- Gladding, T. S., New method of estimating Iron oxide and Alumina in Phosphate rock, 273
 ———, Note on the microscopic detection of Beef fat in Lard, 254
 Glaser, C., Analysis of crude Monazite, and the estimation of Thorium, etc., 274
 ———, F., and Mühle, K., Estimation of Phosphoric Acid in Medicated Wines, 292
 Glass, Influence of Magnesia mixture on, 166
 Glucose, Commercial analysis of, 194
 ———, Estimation of, 238
 Glue, Valuation of, 239
 Glycerin manufacture, Trimethylene Glycol as a bye-product in the, 45
 Glycocol-para-amido-phenetol, 70
 Glycol, Trimethylene, as a bye-product in the Glycerin manufacture, 45
 Goats' Milk, Composition of, 88
 Gold, Determination of, in Pottery Enamel, 140
 Goldenberg's method for Tartar estimation, Modification of, 333
 Gomborg, M., Estimation of Caffeine by means of Wagner's reagent, 193
 Gooch, F. A., and Fairbanks, C., Estimation of the Halogens in the mixtures of their Silver salts, 24
 ———, ———, Pierce, A. W., Iodometric estimation of Selenious and Selenic Acids, 221
 Goose-fat, Composition of, 235
 Goske, A., Emulsified Lard, 45
 Goutal, E., Estimation of Potash by the Carnot method, 216
 ———, ———, Critical examination of the volumetric methods of estimating Nickel, 336
 Gouthière, H., Analysis of Aluminium and its Alloys, 270
 Grape or Invert Sugar, Estimation of, in solutions containing Lead, 46
 Graphite, Estimation of, in Iron, 23
 Green, Brunswick, Analysis of, 230
 Greiner and Freiderichs, Mm., Araeometer Pipette, 249
 Grimwood, R., and Dibdin, W. J., Analysis of Mortar, 197
 Gruber, M., Detection of Ergot in Meal and Bread, 15
 Guano, Estimation of Nitrogen in, 164
 ———, ———, Uric Acid in, 332
 Guaiacol, 296
 Gunpowder, Estimation of sulphur in, 243
 Gutta-percha, Analysis of, 74
 Gutzeit, E., Variation in size of the fat-globules in Cow's Milk, 44
 Haefcke, H., Lösche's process for the estimation of Potassium, 217
 Hahn, J. H., Examination of Sumbul root, 265
 Halogens, Estimation of the, in the mixtures of their Silver salts, 24
 Halphen, C., Detection of Vegetable and Animal Oils in Mineral Oils, 105
 Hansen, E. C., Practical Studies in Fermentation, *Review*, 112
 Hanson, Fortune v., Milk Certificate case, 53
 Hardy, H., and Budden, E. R., Note on the estimation of minute quantities of Metals in Liquids, 12
 Hart, E. B., and Campbell, E. D., Quantitative determination of Hydrogen, 222
 Hattensauer, G., Quantitative Estimation of Arsenic in crude commercial Sulphuric Acid, 166
 Hayes-Campbell, J., and Cushman, A. S., Volumetric estimation of Lead, 48
 Hebert, A., New unsaturated Fatty Acid, 298
 Hefelmann, R., and Mann, P., Examination of Linseed-Oil, Varnish, and Oil-Colours, 46
 Hehner, O., Detection of Formalin, 94

- Hehner, O., Fatty Acids, 173
 ———, Food and Drug legislation, 36
 ———, and Mitchell, C. A., Determination of Stearic Acid in Fats, 316
 Hehner's test for Formic Aldehyde, Note on, 157
 Hempseed Oil, 107
 Henriques, R., Saponification in the cold, 67, 192
 Hensold, O., Extraction of the fat of Cheese for the purpose of examination, 44
 Herbig, W., Estimation of unsaponifiable matter in Wool-fat, 47
 Herlant, L., Electrical conductivity of Fats and Oils, 257
 Hewlett, Dr. —, Typhoid Bacillus in Water, 147
 Hilger, A., and Bauer, F., Examination of Peppers, 257
 Hintz, E., and Fresenius, R., Estimation of Uranium in ores which contain Phosphoric or Arsenic Acids, 26
 Hog's Lard, 326
 Holde, —, Detection of Benzene in Petroleum Spirit, and vice versa, 69
 Holloway, G. T., and Radwood, B., Petroleum, *Review*, 167
 Honey, Contributions to the analysis of, 287
 ———, Microscopy of, 255
 ——— and other substances, Estimation of Lævulose in, 134
 Horse-fat and Horse-flesh, 231
 Horse-flesh, Chemical detection of, 231
 ——— and Horse-fat, 231
 ——— kidney fat, 328
 Howard, Bridge v., Milk Certificate case, 305
 Hughes, J., Mortar, 205
 Hulsebosch, Van L., Valuation of Extract of Cinchona, 47
 Human fat, Composition of, 171
 Hyde, F. S., Scheme for the identification of Acetanilide, Phenacetin, etc., 69
 Hydrochloric Acid, Determination of, in the Gastric Juice, 74
 Hydrocyanic Acid, Detection of, in cases of Poisoning, 160
 Hydrofluoric Acid, Examination of, 243
 ———, Note on the preparation of pure, 87
 Hydrogen peroxide, New reagents for, 80
 ———, Quantitative separation of Metals in alkaline solutions by means of, 79
 Hydrogen, Sulphuretted, Purification of, by means of Iodine, 66
 ———, ——— Arsenical, by Iodine, 236
 Hydrometer, Improved, 304
 Hydroquinone, 296
 Hypochlorites, Chlorides, and Chlorates, Volumetric determination of mixtures of, 221
 Indicator, Resazurine as an, 245
 ——— solutions, Decomposition of Alcoholic, 247
 Industrial Organic Chemistry, Handbook of, 2nd Edn., *Review*, 279
 INORGANIC ANALYSIS. ABSTRACTS, 23, 48, 76, 109, 139, 165, 195, 216, 243, 268, 302, 334
 Invert or Grape Sugar, Estimation of, in solutions containing Lead, 46
 Iodides or Mercury, Nessler's reaction as a test for, 77
 Iodine, Estimation of, in the ashes of Seaweed, 52
 ———, Purification of Arsenical Sulphuretted Hydrogen by, 236
 ——— Sulphuretted Hydrogen by means of, 66
 ——— and Bromine, New reagent for, 68
 Iron, Graphite in, Estimation of, 23
 ———, Manganese in pig, Estimation of, 139

- Iron, Silicon in, Determination of, 53
——— and Steel, Examination of the methods for the determination of Manganese in, 218
——— Titanic Acid, Volumetric determination of, in ores, 77
——— from Beryllium, Separation of, 23
——— ores, Estimation of Zinc in, 335
——— oxide and Alumina, Determination of, in Phosphate rock by the Ammonium acetate method, 272
———, New method of estimating, in Phosphate rock, 273
Isanic Acid, 298
Isonitrile test, 69
Italic. *See* Van Italic
Jandrier and Barbet, Mm., Differentiation of Aldehydes by means of Phenols, and vice versa, 295
———, Estimation of Ethers in Alcohols, 294
Janke, —, Rapid estimation of Zinc in articles of Food, 290
Jannasch, P., Decomposition of Silicates by Boric Acid, 51
———, and Weingarten, P., Estimation of water in Silicates by the Borax method, 109
———, Von Cloedt, E., and Kammerer, H., Quantitative separation of Metals in alkaline solution by means of Hydrogen peroxide, 79
Japan Wax, 193, 330
Jaubert, G. F., Method for distinguishing between Photographic prints produced by development and by direct exposure, 48
Jensen. *See* Otto-Jensen
Jorissen, A., New test for Dulcin, 164
Jüptner. *See* Von Jüptner
Kahlbaum, G. W. A., Liebig Condenser, 84
Kammerer, H., Jannasch, P., and Von Cloetz, E., Quantitative separation of Metals in alkaline solution by means of Hydrogen peroxide, 79
Karlowa, A., and Stutzer, A., Estimation of Uric Acid in Guano, 332
Karsch, W., Leffmann-Beam process for the estimation of Volatile Fatty Acids, 259
Karsten, H., Copper in Wine, 102
Kastle, J. H., New reagent for Bromine and Iodine, 68
Kebler, L. F., Spermaceti, 69
Keller, C. C., Reactions of Digitalin, 46
Kestner. *See* Scheurer-Kestner
Ketel. *See* Van Ketel
Kinder, —, Estimation of Zinc in Iron ores, 335
Kippenberger, C., Estimation of Alkaloids by titration with Iodine solution, 191
Kissling, R., Estimation of Sulphur in Petroleum, 162
Kjeldahl's method and its modifications, 267
Klepszow, N. S., Presence of Solanine in Potatoes, 20
Koch's Sterilizing Cylinder as a Water-bath, 27
Kolanut, Alkaloids of the, 292
———, Analysis of the, 265
Kolanine, 265, 292
König, J., and Bömer, A., Composition of Meat Extract, 17
Koninck. *See* De Koninck
Lactophenin, 291
Lactose, Determination of, in Milk, by double dilution and polarization, 182

- Lævulose, Estimation of, in Honey or other substances, 134
- Langbein, H., Examination of commercial Saccharin by means of the Calorimeter, 264
- Lard, 298, 321
- , Emulsified, 45
- , Hog's, 325
- , Iodine number of, at different times of the year, 15
- , Note on the microscopic detection of Beef fat in, 254
- and Butter analyses, Use of the Calorimeter in, 107
- crystals, 328
- , etc., Notes on, 159
- Oil, 243
- Lasne, H., Natural and industrial Phosphates—III. : the insoluble residue, 246
- , Solution of Phosphates for analysis, 195
- Lead, Electrolytic determination of, and the influence of Arsenic, Selenium, and Manganese thereon, 268
- , Estimation of, Volumetric, 269
- , ———, colorimetrically, 82
- , ——— Grape or Invert Sugar in solutions containing, 46
- , Volumetric determination of, 48
- and Manganese resinate, 262, 300
- chloride, Inversion of Sugar by, 137
- in a sample of Canadian Cheese, 208
- nitrate, Inversion of Sugar by, 137
- resinate, 262
- Ledoux, —, Analysis of Copper and its ores, 51
- Leffmann-Beam process for the estimation of Volatile Fatty Acids, 259
- LEGAL NOTES AND CASES, 53, 305
- Lehmann, K. B., Amount of Copper absorbed by Vegetables from a Coppery soil, 290
- Lemoine, A., Precipitation of Calcium oxalate, 244
- Lenher, V., and Rising, W., Electrolytic method for the determination of Mercury in Cinnabar, 165
- Leonard, N., Note on Hehner's test for Formic Aldehyde, 157
- , and Smith, H. M., Relative composition of Milk, Cream, and skimmed Milk, 283
- Liebig Condenser, 84
- Liebrich, —, Determination of Silicon in Iron, 53
- Ling, A. R., Diastatic power of Malt, 127
- , Method for the determination of the diastatic capacity of Malt, 333
- Linseed, 299
- cake Oil, Examination of, 299
- Oil, 106
- , Varnish, and Oil-Colours, Examination of, 46
- Linton, L. A., Technical analysis of Asphaltum, 161
- Liquids, Note on the estimation of minute quantities of Metals in, 12
- Litmus pencil, 277
- Lohnstein, T., Improved Hydrometer, 304
- Long, J. H., Inversion of Sugar by salts, 136
- Looff, G., Estimation of Morphine in Opium, 163
- Lösche, P., Estimation of Potassium, 81
- Lösche's process for the estimation of Potassium, 217
- Lucas, M., Estimation of Lead colorimetrically, 82
- Lunt, J., Typhoid Bacillus in Water, 146
- Magnesia mixture, Influence of, on Glass, 166

- Maize Oil, 328
 Makin, C. J. S., and Fresenius, H., Estimation of Phenol in Soaps and Disinfectants, 301
 Malt, Method for the determination of the diastatic capacity of, 333
 —, etc., Estimation of the diastatic power of, 122
 Manceau, E., Estimation of Tannin in Wines, 44
 Manganese and Lead resinate, 262, 300
 — in Ferro-Manganese, Inaccuracies in the estimation of, 196
 — Iron and Steel, Examination of the methods for the determination of, 218
 — Pig Iron, Estimation of, 139
 — Steel, Sources of error in Volhard's and similar methods of determining, 334
 — linoleate, 300
 — resinate, 262, 300
 Manganous chloride, Inversion of Sugar by, 136
 — sulphate, Inversion of Sugar by, 136
 Mann, P., and Hefelmann, R., Examination of Linseed-oil, Varnish, and Oil-colours, 46
 Margarine, 258, 321, 328
 Maul, R., and Stutzer, A., Estimation of Fusel oil in rectified Spirit, 213
 McGill, A., Note on the use of the Westphal Balance, 156
 McTaggart, J., Noyes, W., and Craver, H., Determination of the heating effects of Coals, 21
 Meal and Bread, Detection of Ergot in, 15
 Meat Extract, Composition of, 17
 — Extracts and commercial Peptones, Estimation of Gelatin in, 19
 Medicated Wines, Estimation of Phosphoric Acid in, 292
 Meillere, M. G., Improved Molybdate solution, 81
 Meineke, C., Critical examination of the processes for the estimation of Phosphoric Acid, 110
 Mercier, G., Determination of Albumin in Urine, 214
 Merck, E., Molybdic Acid as a test for Alcohol, 163
 Mercuric chloride, Inversion of Sugar by, 137
 — cyanide, Detection of, 165
 Mercury, Detection of, in cases of poisoning, 237
 —, Electrolytic method for the determination of, in Cinnabar, 165
 —, Estimation of, with Sodium Peroxide, 165
 —, General method for the estimation of, 303
 —, Volumetric determination of, by Potassium Iodide, 247
 — or Iodides, Nessler's reaction as a test for, 77
 Meta-dioxy-benzene, 70
 Metallic oxides, Resinated, 261
 Metals, Delicacy of certain tests for, 336
 —, Note on the estimation of minute quantities of, in Liquids, 12
 —, Quantitative separation of, in alkaline solution by means of Hydrogen peroxide, 79
 Methacetic, 291
 Methyl Phenyl Acetamide, 70
 Methylamines, Method for the separation of, 297
 Milk, Asses', Composition of, 88
 —, Boric Acid in, Estimation of, 64
 —, Condensed, Note on the concentration of, 281
 —, Cows', Variation in size of the fat-globules in, 44

- Milk, Fat in, Determination of, 44
 Formaldehyde in, Detection of, 285
 Goats', Composition of, 88
 Preservatives in, Relative efficiency of various, 65
 Skimmed, Cream, and Milk, Relative composition of, 283
 Sodium bicarbonate in, Detection and estimation of, 286
 Sterilization of, 102
 and Butter Certificates, 113
 Milk products, Composition of, 88
 Certificate case. *Bridge v. Howard*, 305
 Fortune v. Hanson, 53
 Cream, and skimmed Milk, Relative composition of, 283
 etc., Tropæolins in, 140
 its Nature and Composition, *Review*, 55
 preservative, Potassium chromate as a, 285
 products and Milk, Composition of, 88
 Milks, Determination of Lactose in, by double dilution and polarization, 182
 Miller, Dr. A. K., *Translator*, Practical Studies in Fermentation, *Review*, 112
 Mineral Oils, 243
 —, Detection of Vegetable and Animal Oils in, 105
 Mitchell, C. A., Composition of Human Fat, 171
 —, Tests for Formaldehyde, 98
 —, and *Hehner, O.*, Determination of Stearic Acid in Fats, 316
 Sykes, W. J., Estimation of the diastatic power of Malt, etc., 122
 —, *W. L.*, and *Wells, H. L.*, Volumetric determination of Titanic Acid and
 Iron in ores, 77
 Moerk, F. X., Detection of Acetanilide, 291
 Moissan, H., Analysis of Aluminium and its Alloys, 83
 Mola, P., Toxicological detection of Aqua Regia, 21
 Molybdate solution, Improved, 81
 Molybdenum, Estimation of, and the volumetric separation of Molybdenum and
 Vanadium, 25
 Molybdic Acid as a test for Alcohol, 163
 — and Vanadic Acids, Reactions of, with Thiocyanates, 273
 Monazite, Analysis of crude, and the estimation of Thorium, etc., 274
 Montpellier, J. A., Analysis of Gutta-percha, 74
 Moor, C. G., and *Pearmain, T. H.*, Bacteriological examination of Water for the
 Typhoid Bacillus, 117, 141
 Moracewski. *See* Von Moracewski
 Morphine, Estimation of, in Opium, 163
 —, Identification of, in toxicological cases, 293
 Morse, H. N., and *Chambers, A. D.*, Method for the standarization of Potassium
 permanganate and Sulphuric Acid, 245
 Mortar, Analysis of, 197
 Motteu, J., and *Crismer, L.*, Saturation temperatures and Critical temperatures.
 Application to general analysis, 241
 Moulin, L., New reaction for Asparagine, 332
 Mühle, K., and *Glaser, F.*, Estimation of Phosphoric Acid in Medicated Wines, 292
 Mustard, Essential Oil of, Estimation of the, in Feeding Stuffs, 233

 Nagelvoort, J. B., Identification of Morphine in toxicological cases, 293
 Nagy Ilosva. *See* Von Nagy Ilosva
 Naphthol, α -, 296
 Action of nascent Bromine on, 238

- Naphthol, α , Ethyl Ester of, Action of nascent Bromine on, 238
 —, β , 296
 —, Action of nascent Bromine on, 238
 —, Ethyl Ester of, Action of nascent Bromine on, 238
 —, Methyl Ester of, Action of nascent Bromine on, 238
 Naphthols and Naphthylamine, Action of nascent Bromine on, 287
 Naphthylamine, α -, Action of nascent Bromine on, 238
 —, β -, Action of nascent Bromine on, 238
 — and Naphthols, Action of nascent Bromine on, 237
 Neats' foot Oil, 243
 Nessler's reaction as a test for Mercury or Iodides, 77
 Neumann, B., Delicacy of certain tests for Metals, 336
 —, Electrolytic determination of Lead, and the influence of Arsenic, Selenium, and Manganese thereon, 268
 Nickel, Critical examination of the volumetric methods of estimating, 336
 — and Cobalt, Separation of, 52
 Nitrites, Cuprous oxide as a reagent for, 246
 —, Determination of, in potable Water, 137
 —, Three new reagents for the detection of, 51
 Nitro-Explosives, *Review*, 277
 Nitrogen, Determination of, in the compounds of Amines with metallic Chlorides, 71
 —, Estimation of, in Guano, 164
 Nitro-Glycerin, Estimation of, in Dynamite, 241
 Noyes, A., and Watkins, W. H. W., Trimethylene Glycol as a bye-product in the Glycerin manufacture, 45
 —, W., McTaggart, J., and Craver, H., Determination of the heating effects of Coals, 21
 Nut Oil, 107
 Nutmeg Oil, 74
 Oetell, F., Estimation of Phosphorus in Phosphor-Bronze, 79
 Oil, Acorn, 74
 —, Almond, 328
 —, Animal, 243
 —, Coconut, 107
 —, Colza, 107, 243
 —, Cotton, 258, 259
 —, —, "Stearin," 328
 —, Cottonseed, 106
 —, Earthnut, 106, 258, 328
 —, —, Colour reaction for, 258
 —, Essential, of Mustard, Estimation of the, in Feeding Stuffs, 233
 —, Fish, Japanese, 243
 —, Fusel, Estimation of, in rectified Spirit, 213
 —, Hempseed, 107
 —, Lard, 243
 —, Linseed, 106
 —, —, Varnish, and Oil-Colours, Examination of, 43
 —, —, -cake, Examination of, 299
 —, Maize, 328
 —, Neats' foot, 243
 —, Nut, 107
 —, Nutmeg, 74
 —, Olive, 73, 107, 258, 259, 298, 328

- Oil, Pine-nut, 74
- , Poppy, 106, 259
- , Rape, 107
- , Rosin-, and Resin, Detection of, in Oils and Varnishes, 164
- , Sesame, 107, 258, 259
- , ———, Reaction for, 135
- , Sheeps' foot, 243
- , Valve, 243
- Oil-Colours, Linseed-Oil, and Varnish, Examination of, 46
- of Bergamot, Examination of, 137
- the Egg, 161
- Oils, Animal and Vegetable, Detection of, in Mineral Oils, 105
 - Californian, Some, 73
 - Determination of the degree of oxidation of, 105
 - heat of Bromination in, 210
 - Essential, Note on a convenient form of Polarimeter for examining, 14
 - Fatty, Estimation of Resin in, 260
 - Industrial, Critical temperatures of, 243
 - Mineral, 243
 - , Detection of Vegetable and Animal Oils in, 105
 - Vegetable and Animal, Detection of, in Mineral Oils, 105
 - and Fats, Bromine heat-value of, 107
 - , Electrical conductivity of, 257
 - Varnishes, Detection of Resin and Rosin Oil in, 164
- Oleomargarine, 108, 321, 328
- Olive Oil, 73, 107, 258, 259, 298, 328
- Opium, Estimation of Morphine in, 163
- Ores which contain Phosphoric or Arsenic Acids, Estimation of Uranium in, 26
- ORGANIC ANALYSIS. ABSTRACTS, 21, 45, 67, 102, 136, 161, 192, 210, 237, 257, 293, 332
- Organic Analysis, Commercial, 2nd Edn., *Review*, 279
- Chemistry, Handbook of Industrial, 2nd Edn., *Review*, 279
- compounds, Estimation of Sulphur in, 46
- Organic salts, Estimation of Zinc in, 334
- Ortho- and Para-Toluidines and Aniline, Estimation of moisture in, 215
- Osborne, T. B., and Campbell, G. F., Chemical nature of Diastase, II., 267
- Otto-Jensen, H., Estimation of Iodine in the ashes of Seaweed, 52
- Oxides, Resinated Metallic, 261
- Oxygen, Determination of, in commercial Copper, 57
- , Estimation of, in Water, 274
- Pade, L., Detection and estimation of Sodium bicarbonate in Milk, 286
- Pajot, —, Adulteration of Rape-seed, 238
- Paladino, P., and Toso, W., Oil of the Egg, 161
- Palmitic Acid, 317
- Para- and Ortho-Toluidines and Aniline, Estimation of moisture in, 215
- Sulphanilic Acid, Estimation of, 214
- Parry, E. J., and Coste, J. H., Commercial Prussian Blue, 225
- Passon, M., Estimation of the Essential Oil of Mustard in Feeding Stuff, 233
- , and Garlach, M., Estimation of available Phosphoric Acid in Basic Slag, 81
- Pastry, etc., Detection of Egg-Yolk in, 233
- Patent Laws of all Countries, Gleanings from the, *Review*, 28
- Pearmain, T. H., and Moor, C. G., Bacteriological examination of Water for the Typhoid Bacillus, 117, 141

- Peinemann, K., Notes on Cubebs, 236
 Peppers, Examination of, 257
 Peptones, Commercial, and Meat Extracts, Estimation of Gelatin in, 19
 Perchlorates, Chlorides, and Chlorates, Analysis of a mixture of, 220
 Persulphates, Analysis of, 139
 Petit, A., and Terrat, P., Estimation of Caffeine in Tea, 232
 Petroleum, *Review*, 167
 ———, Estimation of Sulphur in, 162
 ———, Spirit, Detection of Benzene in, and vice versa, 69
 Phenacetin, 291
 ———, Acetanilide, etc., Scheme for the identification of, 69
 Phenecoll hydrochloride, 70
 Phenocoll, 291
 Phenol, 296
 ———, Estimation of, in Soaps and disinfectants, 301
 ———, salicylate, 70
 Phenols, Differentiation of Aldehydes by means of, and vice versa, 295
 Phenyl acetamide, 70
 ———, Salic, 296
 ———, -dimethyl-pyrazolon, 70
 Phloroglucinol, 296
 Phosphate, Thomas, Analysis of, 132, 151
 ———, rock, Determination of Iron Oxide and Alumina in, by the Ammonium acetate method, 272
 ———, New method of estimating Iron oxide and Alumina in, 273
 Phosphates, Natural and Industrial—III.: the insoluble residue, 246
 ———, Solution of, for analysis, 195
 Phosphor-Bronze, Estimation of Phosphorus in, 79
 Phosphoric acid, Critical examination of the processes for the estimation of, 110
 ———, Estimation of, in Medicated Wines, 292
 ———, ——— available, in Basic Slag, 81
 ———, Gravimetric method of estimating, as Ammonium phosphomolybdate, 219
 ———, and Arsenic Acid, Volumetric process for the determination of, 248
 ———, or Arsenic Acids, Estimation of Uranium in ores which contain, 26
 Phosphorus, Estimation of, in Phosphor-Bronze, 79
 Photographic prints, Method for distinguishing between, produced by development and by direct exposure, 48
 Picric Acid, New reaction of, 332
 Pierce, A. W., and Gooch, F. A., Iodometric estimation of Selenious and Selenic Acids, 221
 Pig's fat, 326
 Pine-nut Oil, 74
 Pipette, Aærometer, 249
 Planchon and Vuafart, Mm., Detection of Borax in Butter, 286
 Platinum, Recovery of, from Potash estimations, 186
 Platt, C., Qualitative examination of Acetanilide, 138
 Poisoning, Detection of Hydrocyanic Acid in cases of, 160
 ———, ———, Mercury in cases of, 237
 Polarimeter, Note on a convenient form of, for examining Essential Oils, 14
 Pope, F. J., Volumetric estimation of Lead, 269
 Poppy Oil, 106, 259
 Potash, Estimation of, 245
 ———, ———, by the Carnot method, 216

- Potash salts, Analysis of, used as fertilizers, 154
 Potassium, Estimation of, 81, 217
 ———, ———, Lösche's process for the, 217
 ——— Aluminium sulphate, Inversion of Sugar by, 137
 ——— chromate as a Milk preservative, 285
 ——— cyanate, Detection of, in Potassium cyanide, 25
 ——— cyanide, Detection of Potassium cyanate in, 25
 ——— dichromate solution, Standard, 82
 ——— permanganate and Sulphuric Acid, Method for the standardization of, 245
 Potatoes, Presence of Solanine in, 20
 Pottery Enamel, Determination of Gold in, 140
 Prager, A., Estimation of Potassium, 217
 Preservatives, Relative efficiency of various, in Milk, 65
 PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS. *See* SOCIETY OF PUBLIC
 ANALYSTS, PROCEEDINGS OF THE.
 Prussian Blue, Commercial, 225
 Public Analysts, 41
 ———, Relations of, to Government Boards, 37
 ——— and Somerset House, 41
 Pyrites ores, Estimation of Pyrrhotite in, 223
 Pyrogallol, 296
 Pyroligneous products, Determination of the acidity of, 211
 Pyrrhotite, Estimation of, in Pyrites ores, 223

 Qualitative analysis, Practical Chemistry and, 6th Edn., *Review*, 28
 Quantitative Chemical Analysis, 3rd Edn., *Review*, 28
 Quinine, Note on the titration of, 85
 ——— sulphate, 70

 Rabbits' fat, Composition of, 135
 Ranwez, F., Application of the Röntgen rays to the analysis of Vegetable sub-
 stances, 233
 Rape oil, 107
 ——— seed, 299
 ———, Adulteration of, 238
 Rebière, G., New method of analysing alkaline Benzoates, 103
 Redwood, B., and Holloway, G. T., Petroleum, *Review*, 167
 Reed, L., Tropæölins in Milk, etc., 140
 Refractometer, Estimation of Alcohol and Extract in Wine by the, 133
 Reichard, A., Silica a cause of Scale in Boilers, 139
 Resazurine as an Indicator, 245
 Resin, Estimation of, in Fatty Oils, 260
 ——— and Rosin Oils, Detection of, in Oils and Varnishes, 164
 Resinated Metallic Oxides, 261
 Resorcin, 70
 Resorcinol, 296
 REVIEWS OF BOOKS. *See* BOOKS, REVIEWS OF
 Richards, E., and Ellms, J. W., Colouring matter of natural Waters, 209
 Richardson, H. A., and Gill, A. H., Determination of Nitrites in potable Water, 137
 Richmond, H. D., Composition of Milk and Milk products, 88
 ———, and Boseley, L. K., Further notes on the detection of Formalin, 92
 Rideal, S., Detection of Formalin, 168
 Riegler, E., Estimation of Alcohol and Extract in Wine by the Refractometer, 133
 ———, Method of estimating Uric Acid, 104

- Rinzaud, M., Artificial Coloration of Wine, 45
 Rising, W., and Lenher, V., Electrolytic method for the determination of Mercury in Cinnabar, 165
 Ritter, G. v., Estimation of Zinc in Organic salts, 334
 Rivière, G., and Bailhache, G., Kjeldahl's methods and its modifications, 267
 Rochefontaine. *See* De Rochefontaine
 Rocques, X., Estimation of Sugar in Chocolate, 256
 Romija, —, Estimation of dissolved Oxygen in Water, 274
 Romijn, —, Reactions of Formaldehyde, 102
 Röntgen rays, Application of the, to the analysis of Vegetable substances, 233
 Rosin Oil and Resin, Detection of, in Oils and Varnishes, 164
 Rozsenyi, J., Composition of Goose-fat, 235
 Ruer, R., Estimation of Potassium, 217
 Rupeau, M., Volumetric determination of Copper, 110
 Rürup, L., Examination of the methods for the determination of Manganese in Iron and Steel, 218
- Sabatier, P., Cuprous oxide as a reagent for Nitrites, 246
 Saccharin, Examination of commercial, by means of the Calorimeter, 264
 Sadtler, S. P., Handbook of Industrial Organic Chemistry, 2nd Edn., *Review*, 279
 ———, Technical analysis of Asphaltum, 211
- Salol, 70
 Salophen, 291
 Sanford, P. G., Nitro-Explosives, *Review*, 277
 Saponification in the cold, 67, 192
 Saturation temperatures and Critical temperatures. Application to general analysis, 241
- Scala, A., Measurement of rancidity of Fats other than Butter, 298
 Scale in Boilers, Silica a cause of, 139
 Scheurer-Kestner, —, Determination of the acidity of Pyroligneous products, 211
 Schiff, F., and Fucks, F., Yellow tint of Zinc-white, 139
 ———, H., Biuret reaction, 103
 Schirmer, M., Determination of Gold in Pottery Enamel, 140
 Schlagdenhauffen, —, Purification of Arsenical Sulphuretted Hydrogen by Iodine, 236
 Schneider, E. A., Detection of Cyanate in Potassium Cyanide, 25
 Schranz, W., and Dobriner, P., Estimation of Aniline and Toluidine in presence of small quantities of each other, 215
 ———, ———, moisture in Aniline and in Ortho- and Para-Toluidines, 215
- Schuyten, M. C., Estimation of Mercury with Sodium peroxide, 165
 Schweinitz. *See* De Schweinitz
 Seaweed, Estimation of Iodine in the ashes of, 52
 Seidel, H., and Ulzer, F., Analysis of Wool-fat, 260
 Selenious and Selenic Acids, Iodometric estimation of, 221
 Sesame Oil, 107, 258, 259
 ———, Reaction for, 135
- Sheeps' fat, 327
 Sheeps' foot Oil, 243
 ——— kidney fat, 328
- Shepherd, H. H. B., Official methods for the analysis of Fertilizers, issued by the German Manure Manufacturers' Association, 99, 128, 151, 186
 Shimer, P. W., Estimation of Graphite in Iron, 23
 Silbermann, H., Estimation of the "Weighting" materials in Silk, 240
 Silica a cause of Scale in Boilers, 139

- Silicates, Decomposition of, by Boric Acid, 51
 ———, Estimation of water in, by the Borax method, 109
 Silicon in Iron, Determination of, 53
 Silk, Estimation of the "Weighting" materials in, 240
 Sinibaldi, J., Estimation of Alcohol in Wine by the Ebullioscope and distillation, 210
 Skraup, Z. H., Purification of Sulphuretted Hydrogen by means of Iodine, 66
 Slag, Basic, Action of Ammonium citrate on, 166
 ———, Estimation of available Phosphoric Acid in, 81
 Smith, E. F., and Atkinson, E., Separation of Iron from Beryllium, 23
 ———, H. M., Note on the estimation of Formic Aldehyde, 148
 ———, and Leonard, N., Relative composition of Milk, Cream, and skimmed Milk, 283
 Soaps, Examination of, 71
 ———, Quantitative analysis of, 72
 ——— and Disinfectants, Estimation of Phenol in, 301
 Society of Public Analysts, Annual Address to the, 29
 ———, ——— Dinner of the, 36
 SOCIETY OF PUBLIC ANALYSTS, PROCEEDINGS OF THE, 1, 29, 57, 85, 113, 141, 169, 197, 225, 309
 Society of Public Analysts, Summer meeting of the, 197
 Sodium bi-carbonate, Detection and estimation of, in Milk, 286
 ——— oxalate, Normal, in volumetric analysis, 336
 Solanine, Presence of, in Potatoes, 20
 Somerset House, Public Analysts and, 41
 Sorel, E., Distillation of the lower Acids of the Fatty series in presence of water, 239
 Sorenden, S. P. L., Normal Sodium oxalate in volumetric analysis, 336
 Source. See De la Source
 Spaeth, E., Detection of Egg-yolk in Pastry, etc., 233
 ———, Examination of Soaps, 71
 Spermaceti, 69, 193
 Spirit, Estimation of Fusel Oil in rectified, 213
 Stahl, K. F., Examination of Hydrofluoric Acid, 243
 Stearic Acid, Determination of, in Fats, 316
 Stearin, 298
 ———, Beef, 328
 ———, Cotton Oil, 328
 Steel and Iron, Examination of the methods for the determination of Manganese in, 218
 ———, Manganese in, Sources of error in Volhard's and similar methods of determining, 334
 Stelling, C., Valuation of Glue, 239
 Sterilizing Cylinder, Koch's, as a Water-bath, 27
 Stevenson, Dr. T., Annual Address to the Society of Public Analysts, 29
 ———, Typhoid Bacillus in water, 147
 Stillman, T. B., Note on the Solubility of Bismuth sulphide in Sodium sulphide, 335
 Stock, W. F. K., Ginger, 311
 Stoddart, F. W., Lead in a sample of Canadian Cheese, 208
 Strohl, A., Iodine number and refractive index of Cocoa Butter, 231
 Stutzer, A., Estimation of Gelatin in Meat Extracts and commercial Peptones, 19
 ———, and Karlowa, A., Estimation of Uric Acid in Guano, 332
 ———, ——— Maul, R., Estimation of Fusel Oil in rectified Spirit, 213
 Sugar, Estimation of, in Chocolate, 256
 ———, Grape or Invert, Estimation of, in solutions containing Lead, 46
 ———, Inversion of, by salts, 136
 ———, Notes on the gravimetric estimation of, by Fehling's solution, 334

- Sulphur, Estimation of, in Gunpowder, 243
 ———, — Organic compounds, 46
 ———, — Petroleum, 162
 ——— and Carbon, Estimation of, in zinc, 76
 Chlorine, Estimation of, by means of Sodium peroxide, 49
 Sulphuretted Hydrogen, Purification of, by means of Iodine, 66
 Arsenical, by Iodine, 236
 Sulphuric Acid, Determination of, or of Barium, 302
 ———, Quantitative estimation of Arsenic in crude concentrated, 166
 and Potassium permanganate, Method for the standardization of, 245
 Sumbul root, Examination of, 265
 Sutton, F., Systematic Handbook of Volumetric Analysis, 7th Edn., *Review*, 308
 ———, W. L., Appointment of, as Public Analyst for Norwich, 28
 Swoboda, A., New reaction of Picric Acid, 332
 Sykes, W. J., and Mitchell, C. A., Estimation of the Diastatic power of Malt, etc., 122

 Tallow, 107
 ———, Mutton, 321
 ———, Stillingia, 328
 Tannin, Estimation of, in Wines, 44
 Tartar, Cream of, Composition and analysis of commercial, 174
 ———, ———, Note on the analysis of, 209
 ———, Crude, and Wine Lees, Estimation of the total Tartaric Acid in, 75
 estimation, Modification of Goldenberg's method for, 333
 Tartaric Acid, Estimation of free, in Wines, 234
 ——— the total, in crude Tartar and Wine Lees, 75
 Tea, Estimation of Caffeine in, 232
 Terrat, P., and Petit, A., Estimation of Caffeine in Tea, 232
 Thiocyanates, Reactions of Vanadic and Molybdic Acids with, 273
 Thiophen, Volumetric estimation of, in Benzene, 297
 Thomas Phosphate, Analysis of, 132, 151
 Thomson, R. T., Estimation of Boric Acid in Milk, 64
 ———, Relative efficiency of various Preservatives in Milk, 65
 Thorium, Estimation of, 274
 Thorne, Dr. T., Relations of Public Analysts to Government Boards, 39
 Thorpe, Prof. —, Public Analysts and Somerset House, 42
 Thymol, 296
 Tiemann, H., Determination of fat in Milk, 44
 Tin, Rapid detection of, 23
 Titanic Acid and Iron, Volumetric determination of, in ores, 77
 Toluidine and Aniline, Estimation of, in presence of small quantities of each other, 215
 Toluidines, Ortho- and Para-, and Aniline, Estimation of moisture in, 215
 Toso, D., and Paladino, P., Oil of the Egg, 161
 TOXICOLOGICAL ANALYSIS. ABSTRACTS, 20, 66, 160, 191, 236, 293
 Trimethylamine, Separation of, from Ammonia, 263
 Trimethylene Glycol as a bye-product in the Glycerin manufacture, 45
 Tropæolins in Milk, etc., 140
 Typhoid Bacillus, Bacteriological examination of Water for the, 117, 141

 Ulzer, F., Analysis of Persulphates, 139
 ———, Detection of Resin and Rosin Oil in Oils and Varnishes, 164
 ———, and Brüll, J., Estimation of Manganese in pig Iron, 199
 ———, ——— Seidel, H., Analysis of Wool-fat, 260
 Uranium, Estimation of, in ores which contain Phosphoric or Arsenic Acids, 26

- Uric Acid, Estimation of, in Guano, 332
 ———, Method of estimating, 104
 ———, Volumetric determination of, 212
 Urine, Determination of Albumin in, 214
 ——— reducing Fehling solution, 334
- Valve Oil, 243
- Van Engelen, A., Colour reaction for Earthnut Oil, 258
- Van Italic, L., Iodine number of Lard at different times of the year, 15
- Van Ketel, B.A., and Antusch, A. C., Examination of Linseed-cake, Oil, 299
- Vanadic and Molybdic Acids, Reactions of, with Thiocyanates, 273
- Vanadium and Molybdenum, Volumetric separation of, 25
- Varnish, Linseed-Oil, and Oil-Colours, Examination of, 46
- Varnishes and Oils, Detection of Resin and Rosin-Oil in, 164
- Vaubel, W., Action of nascent Bromine on Naphthols and Naphthylamine, 237
- Vedrödi, V., Amount of Copper in Vegetable produce, 235
- Vegetable and Animal Oils, Detection of, in Mineral Oils, 105
 ——— produce, Amount of Copper in, 235
 ——— substances, Application of the Röntgen rays to the analysis of, 233
- Vegetables, Amount of Copper absorbed by, from a Coppery soil, 290
- Verbiess, —, Decomposition of Alcoholic Indicator solutions, 247
- Vieth, P., Review of *Milk, its Nature and Composition*, by C. M. Aikman, 55
- Vinegar, White-Wine, 253
- Vitali, D., Detection of Mercuric cyanide, 165
 ———, Mercury in cases of Poisoning, 237
- Voelcker, Dr. J. A., Public Analysts and Somerset House, 43
 ———, Relations of Public Analysts to Government Boards, 37
 ——— and E. W., Note on the composition of two Deposits formed in Water-pipes, 169
- Volhard's and similar methods of determining Manganese in Steel, Sources of error in, 334
- Volumetric Analysis, Systematic Handbook of, 7th Edn., *Review*, 308
 ——— Apparatus, Standardizing, 249
- Von Asboth, A., Estimation of Sulphur in Organic compounds, 46
- Von Cloedt, E., Jannasch, P., and Kammerer, H., Quantitative separation of Metals in alkaline solution by means of Hydrogen peroxide, 79
- Von Dam, W., Determination of Nitrogen in the compounds of Amines with metallic chlorides, 71
- Von Jüptner, H., Inaccuracies in the estimation of Manganese in Ferro-Manganese, 196
- Von Moracewski, W., Determination of Hydrochloric Acid in the Gastric Juice, 74
- Von Nagy Ilosva, L. I., New reagents for Hydrogen peroxide, 80
- Vuaffart, and Planchon, Mm., Detection of Borax in Butter, 286
- Wagner's reagent, Estimation of Caffeine by means of, 193
- Walke and Co., W., Quantitative analysis of Soaps, 72
- Water, Bacteriological examination of, for the Typhoid Bacillus, 117, 141
 ———, Determination of Nitrites in potable, 137
 ———, Estimation of dissolved Oxygen in, 274
 ———, Examination of, 224
 ———, Microscopical, 2
 ———-bath, Koch's Sterilizing Cylinder as a, 27
 ———-pipes, Note on the composition of two Deposits formed in, 169
- Waters, Colouring matter of natural, 209
 ———, Table of analyses and microscopical examination of some, from various sources, 6

- Watkins, W. H. W., and Noyes, A., Trimethylene Glycol as a bye-product in the Glycerin manufacture, 45
- Wauters, J., Reaction for Sesame Oil, 135
- Wax, Bees, 193
- , Carnuba, 193
- , Japan, 193, 390
- Weger, M., Composition of "Driers," 300
- Weingarten, P., and Jannasch, P., Estimation of water in Silicates by the Borax method, 109
- Wells, H. L., and Mitchell, W. L., Volumetric determination of Titanic Acid and Iron in ores, 77
- Westphal Balance, Note on the use of the, 156
- White, Zinc-, Yellow tint of, 139
- Wiley, H. W., Determination of the heat of Bromination in Oils, 210
- , Estimation of Lævulose in Honey or other substances, 134
- , Principles and Practice of Agriculture Analysis, *Review*, 249
- , and Ewell, E. E., Determination of Lactose in Milks by double dilution and polarization, 182
- Wilson, J. A., Bromine heat-value of Fats and Oils, 107
- Wine, Artificial coloration of, 45
- , Copper in, 102
- , Determination of the dry extract of, 66
- , Estimation of Alcohol and Extract in, by the Refractometer, 133
- in, by the Ebullioscope and distillation, 210
- the Volatile Acids in, 158
- Lees and crude Tartar, Estimation of the total Tartaric Acid in, 75
- Wines, Detection of foreign Colouring matters in Red, 289
- , Estimation of free Tartaric Acid in, 234
- , — Phosphoric Acid in Medicated, 292
- , — Tannin in, 44
- Winton, A. L., Modified Ammonium molybdate solution, 220
- Wise, W. L., Gleanings from Patent Laws of all Countries, *Review*, 28
- Wool-fat, Analysis of, 260
- , Estimation of unsaponifiable matter in, 47
- Wright, R. P., and Aikman, C. M., *Translators*, Book of the Dairy, *Review*, 306
- Zamaron, —, Estimation of Grape or Invert Sugar in solutions containing Lead, 46
- Zecchin, M., Modification of Goldenberg's method for Tartar estimation, 393
- Zinc, Acidimetric determination of, 222
- , Estimation of, in Iron ores, 335
- , — Organic salts, 334
- Sulphur and Carbon in, 76
- , Rapid estimation of, in articles of Food, 290
- sulphate, Inversion of Sugar by, 136
- as a precipitant for Albumoses, 16
- White, Yellow tint of, 139

THE ANALYST.

JANUARY, 1896.

EDITORIAL.

WHEN in July, 1891, the Society of Public Analysts re-acquired the copyright of THE ANALYST, and placed the publication of the Journal in the hands of an Editorial Committee, it was with the intention of developing THE ANALYST into a complete and comprehensive record of the progress of analytical chemistry. Twice within the short time which has elapsed since July, 1891, has the Journal been enlarged, partly as regards the number of its pages, partly as regards the quantity of the matter printed on each page. The number of abstracts has increased manifold; and, as far as the space at the command of the Committee would allow, every important paper presenting analytical novelty has been noticed.

The Committee are happy to find that their efforts have met with the general approval of the Members of the Society and of the Subscribers to their Journal.

With the present number commences the twenty-first volume of THE ANALYST. Its size is again increased; twenty-eight pages of matter will be now furnished to the readers in each number, instead of the fourteen which the paper contained at the commencement of its career. Arrangements have been made to collect still more systematically than hitherto everything from chemical literature which the modern scientific analyst should know, if he is to keep in step with the rapid strides of analytical development. It is hoped that THE ANALYST will thus be brought to a state of efficiency worthy of the only Journal exclusively devoted to Analytical Chemistry which is published in the English language.

The Publication Committee rely upon the co-operation of the subscribers in all English-speaking countries, not only for contributions of original matter, but also for the supply of papers containing information which might be usefully abstracted.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, December 4, 1895, at the Chemical Society's Rooms, Burlington House, the President (Dr. Thos. Stevenson) occupying the chair.

The minutes of the previous meeting were read and confirmed.

On the proposal of Mr. A. E. Ekins, seconded by Dr. Voelcker, Dr. F. L. Teed and Mr. John Hughes were appointed to act as Auditors of the Society's accounts for the year.

The following gentlemen were proposed for election. As Member: A. C. Chapman, F.I.C., Analytical and Consulting Chemist. As Associates: Norman Leonard, B.Sc. (Lond.), F.I.C., and Harry M. Smith, assistants to Dr. Stevenson.

The following gentlemen were elected Members of the Society: Thomas Hawkins; Percy Heriot; Frank H. Leeds, F.I.C.; Alex. K. Miller, Ph.D., F.I.C.; C. A. Mitchell, B.A. (Oxon.), F.I.C.; C. H. R. Moore, F.I.C.; Clarence Arthur Seyler, B.Sc. (Lond.), F.I.C.; and Benjamin Watmough.

Dr. BERNARD DYER (Hon. Sec.) read the list of officers for 1896, as proposed by the existing council.

The following paper was read by Mr. Dibdin :

THE MICROSCOPICAL EXAMINATION OF WATER.

By W. J. DIBDIN, F.I.C., ETC.

For some years past it has been the custom for public analysts to include a microscopical examination of the matters held in suspension in water when making an ordinary chemical analysis; but this practice is not universally adopted, and the valuable information which may be so obtained is not infrequently missing in many published results. With the view of placing this important point in its proper position, I venture to submit the following details of the method which I have found to yield unexpected results in my own practice, and which, it is to be hoped, will, after careful trial, be widely, if not universally, adopted.

Hitherto the mode of collecting the suspended matters has been by simple subsidence, often in the bottle in which the sample is collected, but often by allowing the water to stand for some hours in the special settling tubes introduced by Mr. Wynter Blyth, and which I myself employed for a considerable period. These tubes are $2\frac{1}{4}$ inches diameter, and 20 inches long, being contracted at the bottom to a diameter of half an inch. At the bottom of this narrow end, a cup is ground to fit so that the solid matters as they fall are deposited therein. When the subsidence is considered complete, a ground-glass plug, with a glass handle equal to the length of the tube, is placed in position inside the settling-tube just above the cup, when the latter can be removed, and with it the suspended solids, which can then be examined in the usual way.

In the course of a considerable experience with this method, I observed that a small portion of the sediment was collected on the sloping sides of the tube, and all the free swimming organisms did not fall to the bottom, a considerable proportion thus escaping observation. After various trials to overcome these defects, I have adopted a method which removes these objections, and, while rapidly and conveniently enabling the whole of the suspended matters to be placed under one cover-glass on the stage of the microscope, at the same time enables a determination of the volume of the suspended matters contained in a given quantity of water to be recorded. I use the expression "volume" in preference to "weight," as in many otherwise pure waters the total quantity is so small as to be practically unweighable when collected from such a quantity of water as is usually taken for the purpose of analysis; and

again, because the fact of drying so alters the character of the deposit that it no longer represents the condition in which it existed in the water.

By the introduction of the specially-prepared samples of hard filter-paper now so generally employed in the laboratory a ready means is afforded of collecting the sediment by simple filtration, and then washing the so collected sediment into a suitable tube, which I call the "Micro-filter," and in which all the excess quantity of water can be separated, until the whole of the solid matters are contained in one drop of water, thus collecting not only the dead matters, but also the living organisms. This drop of water is then carefully placed on the usual glass slip and examined, first in its natural condition under the microscope, and then, when desirable, after being dried, stained, and mounted in balsam, or water if preferred, for further examination under higher powers for the presence of the various bacteria, etc. One great advantage of this method is that, while the ordinary gelatine plate culture affords valuable information as to the presence of the free bacteria in the water, their presence, often in infinitely greater numbers, entangled with, and feeding upon, the solid matters, is demonstrated in a most striking manner.

The details of the process are as follows: The neck of the bottle containing the sample is first carefully cleansed, to remove extraneous dust, and then one litre of the water, or proportionately less when the quantity of sediment is evidently large, is decanted into a litre-flask, which has been carefully cleansed and washed with pure filtered water. This flask should have a narrow neck, such as those used in the well-known Bischof's bird-fountain, which I have found to be specially suited for the purpose. A clean filter-paper, 3 inches in diameter, is placed in position in a funnel resting in the neck of a flask of at least a litre in capacity, and then the flask containing the sample is inverted so that its mouth is just inside the filter. The water will at once fill the filter-paper, and so close the mouth of the flask, which then acts in the ordinary way of the bird-fountain, feeding the supply in the filter until the whole of the water has passed through. During this operation care must be taken to exclude dust by placing a perforated card, through which the neck of the flask passes, over the funnel. When the water is thus filtered, the deposit is washed from the filter-paper, to which it lightly adheres, into the micro-filter by means of an ordinary wash-bottle having a fine jet and charged with pure water.

The micro-filter is prepared by selecting a length of combustion-tubing, from 8 to 10 inches long, which, after thorough cleansing, is plugged with clean cotton-wool, and then heated to redness in the middle of its length and pulled out to a capillary tube, the narrowest part of which should be something less than 2 millimetres in diameter. It is nicked with a file at the two points at which the tube is exactly 2 millimetres in diameter and neatly broken. By this means two micro-filter tubes will be prepared. They are then converted into filters by charging the small end with a suitable porous diaphragm, prepared by mixing about equal parts of powdered air-dried clay and Kieselghur. This mixture is moistened and worked into a smooth, stiff paste, which is spread out on a slab to the depth of about 2 millimetres. The filter-tube is held perfectly upright and forced steadily down on the mass, and then worked round in small circles. By this method the capillary tube will be neatly charged with a plug of the clay and Kieselghur, which is at once warmed in the

Bunsen flame and gradually heated to redness, so forming a perfect diaphragm admirably suited for its purpose. As the fine sediment from the water will often choke the pores of this small filtering plug, the surplus water may be conveniently removed by putting the small end of the glass tube through an aperture perforated in an indiarubber stopper fitting into a convenient bottle, another tube from which leads to an exhaust pump, which, when worked, creates a vacuum in the bottle, and so pulls the water through the filter. When only about half an inch of water remains the micro-filter is removed, and the depth of deposit which will now be collected on the filter-plug is measured, and the results expressed in terms of millimetres of deposit in a 2-millimetre diameter tube per litre of water. Thus, a numerical factor is obtained to express the actual quantity of suspended matter present in the water; of course, in the case of dirty water, when the deposit from a litre would be too great to be contained in that part of the micro-filter-tube having a diameter of about 2 mm., half or a quarter of a litre may be taken, or even less, and the depth of deposit actually found should be multiplied accordingly, so that the results in all cases, even the most foul waters, where only a few c.c. of the water can be used, may all be expressed in the same terms. So delicate is this method that I have repeatedly demonstrated the presence of most objectionable matters contained in a single tumblerful of so-called ordinary good drinking-water.

When the depth of the deposit has been thus recorded, the filter-tube is scratched with a sharp file about half an inch from the filter-plug and broken off. To remove the deposit which will often be found closely adhering to the plug, a fine, clean platinum wire is pushed up the tube, and used to loosen the fibrous sediment and pull it away from the plug. Upon inverting the tube for a few moments, the sediment will fall to the open end. The tube is cut again with the file close to the plug and the plug end broken off, the tube being held horizontally the meanwhile. We now have the deposit contained in the minute quantity of water held by capillary attraction in the tube thus open at both ends. On holding the tube upright over the microscope glass slide, and giving it a gentle downward shake, the deposit will fall down with one drop of water, and may then be at once covered with a thin glass, and examined preferably at first with a low power, and then with higher powers as may be found desirable. I venture to predict, from experience with various operators, that no little astonishment will be expressed by those who have employed the usual methods at the unusually large quantity and varied character of the deposit so obtained from waters which are constantly passed as containing no suspended matters. Notably the presence of decomposing organic substances swarming with various kinds of bacteria will probably be the most striking when once the observer has accustomed himself to the method of finding them. Many observers are under the impression that these minute organisms cannot be readily seen with the quarter-inch objective usually employed. This is an entire mistake. The only precautions necessary are to work with the critical image, and to focus carefully the minute gelatinous specks which will be often seen close to or along the edge of particles of *débris*. When such are observed, the employment of the D eye-piece will bring them out distinctly, provided the object-glass be properly corrected for the cover-glass, and no other power will be required to differentiate bacilli from micrococci or diplococci. If it is desired to

proceed still further, the use of an oil-immersion lens of as wide a numerical aperture as is obtainable will be necessary, when, especially if the preparation is stained by one of the usual methods, the bacteria will stand out boldly. How far direct microscopic examination may enable a skilled observer to differentiate between pathogenic and non-pathogenic organisms remains for the future to show. Up to the present we can only say that such and such types are present in greater or less numbers, and this without the lengthy process of the gelatine-plate cultures, but which, nevertheless, afford a doubtless valuable aid to the diagnosis of the quality of a drinking-water. By the combined results of that method with the one which I have described, aided by the ordinary chemical examination, no careful analyst could possibly certify as pure water contaminated with objectionable matters, and it may be confidently asserted that, whatever elements of doubt may have more or less legitimately existed during the past as to the value of an ordinary analysis of water, the question has now entered upon a new phase, and is becoming more nearly based upon the foundation of exact knowledge.

Before any results are accepted, care should be taken to make a series of blank experiments with the purest water obtainable, in order that the accidental errors due to dust, etc., may be known and allowed for. A certain minute quantity of fibre will be washed from the filter-paper, and I have found starch granules from the same source. The character of these accidental contaminations is so marked, and essentially different to the objectionable matters found in more or less foul waters, that the careful operator cannot fail to differentiate between them. It will be evident that a few clean linen fibres, even with an odd leguminous starch grain, are entirely different to the decomposing matters so often met with.

In the table attached I give a series of typical results, with the full analyses in each case, in order that the effect of the method may be clearly seen.

They are placed in order of the source of the water, viz., deep and shallow wells, rivers, and brooks, in drought and in flood, etc., and well show the variation in the quantity and quality of the suspended matters as determined by the extremely rapid and exact method I have ventured to submit to the criticisms of the society.

In connection with the microscopical work, I may call attention to the special stand constructed by Messrs. Powell and Lealand, at my request, for the purpose of facilitating the counting and examination of the colonies obtained on gelatine-plate cultures. The stage has mechanical movements to the extent of 4 inches, and is arranged to support the ordinary glass culture plate generally employed. When the colonies are viewed with dark ground illumination, by means of a 4-inch object-glass and binocular vision, the process of counting proceeds with comfort and exactness, as well as greater rapidity.

DISCUSSION.

Mr. WYNTER BLYTH referred to the method of Dr. Klein in searching for the typhoid bacillus, which consisted in filtering a litre of water through a Pasteur candle and then cultivating the sediment. He also described the method of filtration of organisms in use by the biologist of the Massachusetts Board of Health. He considered that it would be better to state the results in cubic millimetres, which

WATER

Table of Analyses and Microscopical Examination
All quantities are stated in grains per gallon, except organic

SOURCE OF WATER.	EXAMINATION IN A 2-FOOT TUBE.				Phosphoric Acid.	AMMONIA.		Chlorine.	OXYGEN ABSORBED IN		Nitrogen as Nitrates, etc.
	Appearance.	Colour by Lovibond's Tintometer Scale.				Free.	Albumi- noid.		Fifteen Minutes.	Four Hours.	
		Red.	Yellow.	Blue.							
Chalk well	clear	colourless			V. S. T.	0.0001	0.0014	1.55	0.010	0.019	0.370
Chalk well	clear	colourless			V. S. T.	0.0001	0.0088	1.70	0.014	0.029	0.870
Chalk well	clear	colourless			none	0.0000	0.0021	1.75	0.017	0.021	0.845
*Deep well	—	—			abundant	0.0220	0.0880	2.50	0.057	0.091	0.540
*Deep well	—	—			traces	0.0030	0.0010	1.60	0.014	0.061	traces
†Deep well, 60 ft. deep	—	—			—	traces	0.0010	5.65	—	0.027	1.010
Deep well, in clay ...	clear	colourless			V. S. T.	0.0000	0.0080	2.10	0.018	0.030	0.140
*Spring, on side of hill	—	—			consider- able trace	0.0110	0.0340	1.80	0.202	0.436	trace
†Spring, turbid after heavy rains ...	—	—			—	0.0020	0.0010	1.25	—	0.027	0.028
†Spring	—	—			—	0.0000	0.0000	0.50	—	0.020	—
*Well	pale greenish	slightly turbid			V. H. T.	0.0030	0.0020	6.00	0.012	0.024	1.88
*Well	yellowish	turbid			traces	0.0010	0.0020	7.60	0.012	0.027	0.16
†Well	—	—			—	0.0000	0.0050	2.70	—	0.040	0.29
*Well	yellowish	turbid			V. H. T.	0.0010	0.0031	9.20	0.197	0.337	4.36
*Rivers	—	—			heavy traces	0.0060	0.0070	3.80	0.084	0.116	traces
*Rivers	—	—			traces	0.1090	0.0250	382.50	0.160	0.289	traces
River in drought ...	slightly turbid	1.0	2.1	—	slight trace	0.0010	0.0130	1.30	0.057	0.092	0.180
River in flood	very turbid	2.5	4.8	—	heavy trace	0.0060	0.0360	1.50	0.114	0.295	0.080
Filtered river water in drought	clear	0.8	0.7	—	—	0.0001	0.0056	1.25	0.035	0.058	0.188
Filtered river water in flood	clear	0.4	0.9	—	S. T.	0.0007	0.0091	1.50	0.057	0.109	0.144
†Brook, above sewage outfall	—	—	—	—	—	traces	0.0030	2.50	—	0.152	0.150
†Brook, below sewage outfall	—	—	—	—	—	0.8000	0.1500	8.75	—	0.541	0.010
†Unknown	—	—	—	—	—	traces	0.0010	1.85	—	0.028	1.290
†Shallow well, 14 ft. deep	—	—	—	—	—	0.0020	0.0040	11.75	—	0.084	1.150

In each case the examination of suspended matters was made by the author. The chemical
Dr. J. A. Voelcker and Mr. E. W. Voelcker.

ANALYSES

of some Typical Waters from Various Sources.

carbon and nitrogen, which are stated in parts per 100,000.

ORGANIC ELEMENTS.			Total Solids.	Cultivation tests by Gelatine Plates.	SUSPENDED MATTERS.	
Carbon.	Nitrogen.	Total.			Mm. of deposit in micro-filter from 1 litre.	Microscopical Examination.
0.055	0.010	0.065	28.00	39	trace	Few infusoria, fibres, and débris.
0.078	0.024	0.102	81.40	76	trace	Various fibres (some dyed), diatoms, and little débris.
0.050	0.007	0.057	26.20	38	0.5	Few fibres (some dyed), general organic débris, bacilli, and micrococci.
—	—	—	—	—	2.5	Black flocculent, spiral vessels, infusoria (paramecium), scale of lepidoptera, animal feathery hair, brown vegetable cellular tissue, dyed cotton-fibres, etc., spongy parenchyma, amœbæ, numerous particles of organic matter (some gelatinous in appearance, swarming with bacilli, micrococci, and diplococci), fungoid mycelium, (?) beggiatoa.
—	—	—	34.16	—	1.0	Fine mineral débris, confervæ, algæ, streptothrix, various small infusoria.
—	—	—	42.28	—	1.7	Cotton-fibre, dyed wool-fibre, infusoria, vegetable epidermis, quantity of mineral matter.
—	—	—	35.20	—	trace	Dyed fibres, anguillulæ, fungoid mycelium, and yellow red stained tissues, organic débris, with bacilli, micrococci, etc.
—	—	—	21.84	—	4.0	Infusoria, diatoms, few fibres, algæ, numerous bacilli and micrococci, débris, etc.
—	—	—	8.60	—	24.0	Mineral and organic débris, rotifers and other infusoria, fungoid mycelium, diatoms, algæ, anguillulæ, sphaeroplea, spongy parenchyma, colonies of bacilli, micrococci, and diplococci, cotton-wool and other fibres (some dyed).
—	—	—	2.24	—	1.5	Brown flocculent, blue-dyed wool and other fibres, gelatinous masses infested with bacilli, micro- and diplococci, fat globules, fragment of insect.
—	—	—	43.96	1250	12.0	Filamentous cellular growth, animal hair (? mouse), dyed wool-fibre, rotifer, diatoms, desmids, potato starch.
—	—	—	20.16	250	15.2	Ferruginous (mostly fine granular matter, with some bacilli and micrococci).
—	—	—	17.36	—	10.0	Quantity of finely-granulated matter, cotton-fibre, vegetable epidermis, fungoid mycelium, few bacilli.
—	—	—	62.16	8500	10.4	Dyed and undyed wool, and cotton fibres, numerous moving organisms, confervæ, fungoid mycelium, numerous bacilli and micrococci.
—	—	—	29.12	—	16.0	Brown mud, quantity fine mineral débris, numerous scales of insects, vegetable débris, etc.
—	—	—	670.60	—	27.0	Brownish-gray mud, confervæ, diatoms, various infusoria, vegetable tissues, oscillaria, anguillulæ, amœba, etc.
0.170	0.085	0.205	19.00	6120	3.0	Usual river débris, larvæ of insects, diatoms, coloured fibres, etc.
0.424	0.053	0.477	29.40	10200	84.0	River débris, with numerous diatoms, fragments of insects, fibres (cotton, wool, etc.).
0.119	0.048	0.162	17.40	19	0.5	Fibres and organic débris, with very numerous bacteria, etc.
0.215	0.060	0.275	23.7	153	1.0	Few fibres (some dyed), living animalculæ, decaying matter, starch cells.
—	—	—	29.40	—	17.0	Mineral débris, oxide of iron with brown filaments, confervæ, vegetable débris, diatoms.
—	—	—	37.80	—	18.0	Quantity fine granular débris with few fibres, the whole swarming with various infusoria and bacteria, etc.
—	—	—	81.90	—	11.5	Brown mud, parenchyma, and other vegetable tissues, fragments of insects, confervæ, etc.
—	—	—	78.10	—	10.4	Cotton and wool fibres (some dyed), few infusoria, quantity oxide of iron, woody fibre, few diatoms, mineral débris.

analyses of samples marked * were made by Dr. Bernard Dyer, of those marked † by In the other cases by the author.

would enable the size of the tubes to be varied. He was confident that such a method of filtration as described by Mr. Dibdin was of the highest value. It was now a question whether it would not be advisable to perform analyses always on the filtered water. To translate potato starch, diatoms, desmids, and so forth, into carbon and nitrogen was but of little utility in comparison to the identification of the organisms themselves, and of the nature of the deposit.

Dr. VOELCKER observed that, while determining the quantity of deposit obtained, it was necessary to bear in mind also the *nature* of the deposit, and this to a certain extent influenced the interpretation of its quantity. In dealing with an ordinary town supply, the condition of which was pretty constant, it was fair enough to regard the deposits obtained from different samples as quantitatively comparable; but in many cases it was greatly a matter of chance what quantity and what kind of deposit was found in the samples. In fact, a sample might be quite different, both as to chemical characteristics and as to suspended matter, from the supply whence it was taken.

Dr. RIDEAL asked whether Mr. Dibdin could give any figures as to the free and albuminoid ammonia in water which had been subjected to his process of filtration, as compared with water that had not been so treated. It seemed to him that a few millimetres of this suspended matter (which would probably contain a good deal of matter, the nature of which was not nitrogenous) would not seriously affect the amount of ammonia present.

Mr. D. A. SUTHERLAND said that to facilitate the counting and separation of the colonies he considered it preferable to attenuate 1 c.c. of the water by mixing it with 10 c.c. of gelatin broth, and again attenuating 1 c.c. of this mixture with another 10 c.c. of broth, and so on. Dr. Woodhead had recommended this plan to him, and he had found it to answer very well. He considered that any examination from a bacteriological point of view of a sample of water received in the ordinary way could be of little or no value. It was essential, especially when the colonies were to be counted, that their cultivation should be commenced at the moment when the sample was actually drawn. He noticed that the deep well water referred to in the table appeared, from its analytical results, to be much worse than the water from the shallow well. He would like to inquire the depth of the so-called deep well, as in his experience he had found wells only thirty or forty feet in depth termed deep wells. The lining of such a well was frequently defective, allowing of surface contamination from a wide area, and this class of well could not be too severely condemned.

Mr. HEHNER thought that too much importance ought not to be attached to the mere number of organisms in the sediment. Water from the purest of mountain streams contained multitudes of diatoms, which, in fact, were rather an indication of purity than otherwise. With regard to the question of uniformity in the statement of results, the Society had, as Mr. Dibdin had pointed out, made great and successful efforts in this direction. Up to the time when the Society's Water Committee took the question up, every chemist determined what he pleased, and returned his results in any notation that was convenient to him; but the great majority of English analysts, and many on the Continent and in America, now worked upon one uniform plan. Now, however, that decimal systems of notation were being so widely introduced, it would, he thought, be a great improvement if the results of water analysis were stated in decimal notation.

Mr. JOHN WHITE said it seemed to him that the filtration of the water through paper was a weak point. Most of the papers of the class referred to by Mr. Dibdin would pass barium sulphate in suspension, so that it was pretty certain that micro-organisms would go through, especially if the papers happened to contain what were technically known as "pin-holes." For collecting the suspended matter, however, for subsequent microscopical examination, this method was certainly the best that had hitherto been proposed. He did not quite understand how Mr. Dibdin had arrived at the figure 84.5 mm. given in the table. If he had drawn out a tube 84 mm. long, and of a uniform diameter of 2 mm., it was a most remarkable feat of dexterity in glass manipulation.

Dr. WASHBURNE said that he had known bacteria pass through even a Berkefeld filter, owing probably to some slight crack. As the morphology of bacteria varied very greatly under different conditions, it was not possible to identify them completely by microscopical examination alone. With regard to counting the number of colonies, he thought it best to so dilute the water that the colonies were sufficiently separated and of such a size as to be easily recognised, either by the naked eye or with the aid of a small hand lens.

Mr. CASSAL said that some persons regarded the microscopical examination of water as a matter of very minor importance. His own opinion was that a microscopical examination carefully carried out afforded most useful evidence, and was, in fact, an adjunct of the utmost value to the chemical part of the analysis. From his somewhat extensive experience in the analysis of water, having been constantly engaged for a number of years in analysing waters, he felt sure that a water analysis for sanitary purposes could not be regarded as satisfactory if it did not include a proper microscopical examination, even in the case of waters the general characters of which were well known. Mr. Dibdin's process was specially applicable, and was specially valuable in the case of a water containing small quantities of suspended matter; and he thought that the results which had been detailed were of much interest and importance. Since Mr. Dibdin had been good enough to show him his method of working, he (Mr. Cassal) had applied it to a number of samples of water with very satisfactory results, and he had found that he was largely helped by it in forming his opinions on the samples. The process was not intended to separate out all the micro-organisms—meaning by this term the "bacteria"—which might be contained in a water, and he did not understand that Mr. Dibdin had intended it for any such purpose; but the process nevertheless arrested what he might call a "sufficiency" of such organisms when they were present in the suspended matter. Of course, with waters which contained considerable quantities of suspended matter, a sufficient amount could be obtained for examination by allowing settlement in suitable apparatus, and for this purpose he had long been accustomed to use the ingenious settling-tube introduced by Mr. Wynter Blyth, with the slight modification of a glass-tapped cup or separator fitted to the end of the tube, which allows a drop to be taken out at will on to a slide. Although this method was sufficient when dealing with comparatively large amounts of suspended matter, which could easily be obtained by subsidence, Mr. Dibdin's practice of measuring the volumes of sediment obtained obviously yielded useful indications, if those indications were considered intelligently; and that this was

so was testified to by the fact that, even some members who had spoken a little disparagingly of Mr. Dibdin's experiments in this direction, were nevertheless anxious to have further information on the point. Allusion had been made to certain so-called experiments carried out several years ago, and reported in the eleventh annual report of the Local Government Board for 1881-82. These "experiments" consisted mainly in the haphazard addition, without proper mixture, of small amounts of urine, excremental matter, and infected liquids, to samples of water, and in then submitting these to certain chemists for analysis. He (Mr. Cassal) had had occasion to look into these "experiments," and in a paper written by Dr. Whitelegge and himself most of the fallacies and absurdities in this report had, he believed, been called attention to and exposed. The report in question, except in one respect, was worthless, and the experiments it detailed were ludicrous; but it was still occasionally referred to as if it contained information of value. The point in regard to which the report was of interest was that some of the infected samples had been examined microscopically, and the microscopic results were amply sufficient to absolutely condemn them. With regard to the bacteriological examination of water, Mr. Cassal again felt it necessary to point out that far too much importance was being attached by some people to the present value of bacteriological processes in water analysis. The mere counting of colonies afforded but an additional test of a rough and misleading character for the existence of pollution; and as to the detection of specific pathogenic micro-organisms, in no single instance in which an outbreak of disease could be attributed to drinking polluted water had bacteriologists been able to assert that they had, with any certainty, identified those micro-organisms which would produce the disease in question. On the other hand, having regard to present bacteriological methods and knowledge, it would be foolish to pass a water as safe upon negative bacteriological results.

Dr. PAKES observed that if Mr. Cassal had read the last Local Government Board report he would be aware that Dr. Klein had, in the case of one of the last two epidemics of typhoid, been able to identify the typhoid bacillus in the water. He (Dr. Pakes) had himself succeeded in absolutely determining the presence of the typhoid bacillus and the bacillus coli communis by bacteriological methods in samples of water that had been passed as pure by analytical chemists.

Mr. CASSAL had not seen the particular report referred to by Dr. Pakes; but he presumed that that report was like others that he had seen, in which the investigator stated that he had found a micro-organism which he was unable to distinguish from the typhoid bacillus. This was very different from any statement to the effect that the typhoid bacillus had actually been identified, and that it was a bacillus which would produce typhoid.

Dr. DYER desired to thank Mr. Dibdin for having kindly examined for him by this process a considerable number of water samples. He had himself tried this process of separation, and concurred in all the remarks that had been made as to its ingenuity, and as to its probable usefulness in enabling the ordinary microscopical examination of the grosser particles of suspended matter in water to be made with more exactness than had hitherto been possible. At the same time, he had compared Mr. Dibdin's method with that of subsidence in the old-fashioned conical glass, and found that, although the new process was undoubtedly more delicate, the results,

qualitatively speaking, were substantially the same. With regard to the quite separate question that had been raised about the distinction between pathogenic and non-pathogenic organisms in bacteriological water examination, he thought that there were really very few cases in which there was any necessity, even if it could be done with facility, to look for or demonstrate the existence of special pathogenic organisms in water. Cases did occasionally arise, no doubt, in which it was desirable not merely to know that water was contaminated, but to trace the introduction of a specific disease, and under some circumstances it might be possible to do it. As a general rule, however, the main point was to demonstrate the presence of sewage. If sewage was present, it mattered little whether pathogenic organisms were or were not present at a given moment, since the water must in any case be condemned. With regard to bacteriological examination, he thought that those analysts who had not familiarized themselves, to a certain moderate extent, at any rate, with bacteriological work, were apt to somewhat under-estimate the value of simple bacteriological methods as aids to the formation of a correct deduction from the chemical results. Sometimes the chemical results were just a little doubtful or suspicious; and it very often happened in cases of that kind that a bacteriological examination yielded results which were of very great assistance to the analyst. It was desirable to make not merely gelatine plate cultivations at the ordinary temperature, but also agar-agar cultivations, with and without the addition of phenol, at blood heat.

The PRESIDENT said that this method had been brought under his notice by Mr. Dibdin some months previously, and he had found it very advantageous. Criticisms had been passed upon the mode of measuring the amount of the deposit, but seeing that the object was to measure merely the bulk of the suspended matter, and not its weight or absolute amount, he thought that Mr. Dibdin's method would probably answer all ordinary purposes better than previous methods.

Mr. DIBDIN said that there was no difficulty in cutting off the tube of the micro-filter at a point where its diameter was exactly 2 mm. He had found that this was about the most convenient for practical work. It was of course necessary to adhere to one fixed diameter in order to obtain comparable results. The measurement of the depth of the sediment was only relative, and not absolute, in consequence of its often flocculent character. Dr. Voelcker's remarks pointed to the desirability of the samples being collected by the analyst himself, or by a careful and specially-trained assistant. He (Mr. Dibdin) did not attach quite so much importance as Mr. Sutherland did to the bacteriological examination being started at the moment of taking the sample. The main point was that the sample should be a fair one, and free from any extraneous matter. The fact of the deep well water showing more objectionable features than that from the shallow well might easily be accounted for by the existence of a fissure in the chalk, which, if it extended into the neighbourhood of a cesspool, would offer a very probable explanation. Such points as this were of course liable to arise in individual cases; no universal rule could be laid down. The figure 84.5 mm., alluded to by Mr. White, had been obtained by calculation from the results of experiments upon a much smaller quantity than the one litre to which the figure referred. Dr. Washburne seemed to think that there was no need for microscopical examination if only the water was sufficiently diluted in making the

gelatin-plate cultivations. He (Mr. Dibdin), however, had found from his own experience that in practice the microscopical examination was of the greatest value, as it not only indicated the presence of bacteria, but the character of the sediment. The microscope was one thing and plate-cultures another, and both should be employed. With regard to Dr. Dyer's remarks as to comparison between the conical-glass method and the use of the micro-filter, he might mention that in the case of the latter a water could be prepared for microscopical examination in half an hour's time, whereas, in the case of a sedimentary process, twenty-four hours elapsed before the examination could be made, which even then was only partial, and took no account of the quantity.

NOTE ON THE ESTIMATION OF MINUTE QUANTITIES OF METALS IN LIQUIDS.

BY E. RUSSELL BUDDEN AND H. HARDY.

(Read at the Meeting, November 6, 1895.)

IN continuance of our experiments on the estimation of minute traces of metal in liquids, we tried the effect of various substances in common use in the preparation of beverages, to see if they produced any appreciable alteration of the tints in colorimetric testing. In addition to sugar, which has much the same effect as glycerin in preventing precipitation and deepening colour in many cases, we tried the effect of essence of lemon, saccharin alone, and saccharin and sugar (as employed in the manufacture of lemonades), but the results obtained were of a negative character, and would certainly have little, if any, practical effect upon results hitherto recorded, since the alteration of tint is, in nearly every instance, too slight to be appreciable. We noted, however, that in the case of the estimation of iron the tint rapidly darkens if the solution be allowed to stand for any length of time—a further indication of the importance of making these tint-tests under precisely similar conditions in every way.

Continuing our search for accurate methods of metal determination, we made a large number of experiments on the deposition of metals from solution by electrolytic processes. In the case of copper, the results obtained were fairly satisfactory, the necessary conditions apparently being that the current must be continued for a long time, and the film upon the platinum electrodes very carefully washed with alcohol and thoroughly dried. With all precautions, however, we found the tendency of the results was to be somewhat low.

With lead, however, the results obtained were extremely unsatisfactory. Theoretically it would seem that the lead should be deposited upon the positive electrode. But, in varying degree, owing apparently to differing degree of acidity of solution, this metal deposits upon both electrodes. The metal is deposited in the form of oxide, and the proportion upon the opposite electrodes is not uniform. There is a great tendency for one or other of the deposits to redissolve, and we only obtained accurate results in about one-third of the determinations made; the results are usually too low. The process at present does not give very satisfactory results when

copper and lead are both present in a liquid, owing to the irregularity of behaviour of the lead.

With mercury, if the solution be moderately strong, the metal is rapidly deposited upon the platinum plates and falls off in globules; in weaker solutions the globules aggregate chiefly upon the plates. In the case of weaker solutions a curious divisional line forms in the solution exactly at the level of the lower edge of the positive electrode. This line becomes violently agitated immediately before the falling down of a globule.

We have not yet been able to effect satisfactory separation of minute traces of metals when several are present in a liquid, and upon the whole find the colorimetric methods preferable for attaining this end in the testing of beverages for metallic contamination.

DISCUSSION.

The PRESIDENT said that in cases where the metals to be estimated existed in presence of organic compounds it was necessary to know the condition of the organic matter, in order to be certain of success and with colorimetric methods. Otherwise the organic compounds must be destroyed and the metal redissolved—a process not always possible in cases where the metal was present only in very small quantity.

Mr. BEVAN said that in recently making some estimations of lead in mineral waters he had found comparison with a simple aqueous solution to be comparatively useless.

Mr. ALLEN said that he had had occasion to examine many thousands of samples of water for lead. Under favourable conditions, the depth of colouration produced by sulphuretted hydrogen was fairly proportionate to the amount of lead present, but where the water contained much mineral or organic matter he thought the results thus obtained were unreliable. Sugar and other forms of organic matter caused lead sulphide to assume a colloidal form, so that the liquid appeared of a brown colour, and no precipitate could be removed by a filter. A better plan was to work on a solution of the ash. It was very important to avoid free acid, as the formation of minute traces of sulphide of lead was much impeded by acids, even acetic acid. When water was derived from recently-melted snow it caused an immediate decomposition of sulphuretted hydrogen, with precipitation of sulphur—a fact which Mr. Allen suggested was due to the presence of peroxide of hydrogen in the snow-water. Mr. Allen considered that for many purposes potassium bichromate was preferable to sulphuretted hydrogen as a test for lead. He allowed a drop or two of a saturated solution of bichromate to fall, without agitation, into the water contained in a tall glass cylinder. As the heavy liquid fell through the water the chromate of lead produced a yellow milkiness, which was very characteristic. The effect was instantaneous with one-twentieth grain per gallon, and with smaller quantities if more water was employed and time allowed. Some years ago all the water supplied to his house contained half a grain of lead per gallon, and he recovered the impurity from a quantity of the water in the form of an ingot of metallic lead by precipitating the metal as chromate and reducing the lead from the precipitate.

NOTE ON A CONVENIENT FORM OF POLARIMETER FOR EXAMINING ESSENTIAL OILS.

By E. RUSSELL BUDDEN.

(Read at the Meeting, November 6, 1895.)

IN the chemical and physical examination of many of the essential oils in use for flavouring purposes, the rapid determination of the specific gravity and optical rotation of the sample greatly facilitates the forming of an opinion as to purity, and not infrequently obviates the necessity of an elaborate chemical analysis. But the ordinary appliances of these determinations are cumbrous and costly. For the specific gravity, however, it is now possible to obtain a very inexpensive and sufficiently accurate plummet-balance, after the fashion of the Westphal, but much cheaper, and just as good. Until recently I have not found a really satisfactory and inexpensive polarimetric instrument, however. After trying various polarimeters, I found that E. Leitz, of Wetzlar, had devised a very beautiful little instrument intended for pathogenic work in the testing of diabetic urine and similar matters. It seemed to me that this possessed so many advantages over the larger and more costly instruments that I made a number of experiments with it. It seemed, however, that the original form of the apparatus was too restricted as to the scale for work of the class I required, since the rotation of some oils is very high. I therefore communicated with the maker (through his London agents, Messrs. C. Baker, of 244, High Holborn), and ultimately obtained an instrument with a complete divided circle. This is as perfectly constructed as any I have ever seen, and the arrangement for rotation and reading of the scale is very convenient indeed. One special advantage of the instrument is that it can be inclined at any angle and used with ordinary light. A polarimeter requiring monochromatic light is, in many small laboratories, a great inconvenience. The description of the optical and mechanical arrangements, given by the maker, is briefly as follows: The improved Mitscherlich consists of two Nicol prisms, between which the object-tube is fitted. The polarizer is fitted upright. Behind the polarizer is the bi-quartz plate, turning to right and left. The analyser, with a vernier attached on a radial arm, traverses the divisions on a circular plate by means of an endless screw. A small movable mirror illuminates the apparatus, and therefore the latter can be brought into any position for comfortable working. The vertical crossed position (*viz.*, entire darkening of the visual field) corresponds to the zero-point. As this position cannot be again obtained after the introduction of an optically active substance, because of the rotation of the latter, the colour—violet—has been chosen as the end-point; the transition of this colour into blue on the one side and into red on the other can be observed most exactly. The range of prismatic colours is seen to be repeated on turning. As long as blue is seen the vernier has not passed far enough; but when red is seen it has passed too far. The eye-piece is movable, and can be focussed exactly on the bi-quartz plate. The particular fraction of a degree that can be read depends on the experience and colour-sensitiveness of the operator. There is a convenient arrangement for adjusting the

zero-point, and although, of course, there is a personal factor in all these observations, I have found it easy to read to one-tenth of a degree with this instrument. I strongly recommend it to those who have occasion to do work of this class.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD ANALYSIS.

The Detection of Ergot in Meal and Bread. M. Gruber. (*Arch. Hyg.*, 1895, xxiv., 228; through *Chem. Zeit. Rep.*, 1895, 329.)—Contrary to the experience of most observers, the present author finds microscopic examination of bread, etc., better adapted for the detection of ergot than the chemical methods. In the case of meal, Vogel's and Hoffmann's colour reactions, nevertheless, succeed perfectly, and they are also available for bread, provided the ergot is present in considerable quantity. For detection by means of the microscope, a little of the flour, or a few crumbs of the bread, are moistened with water, placed on a slide, the cover applied, and the mass heated to the boiling-point. In this manner the starch granules are sufficiently swollen to permit the broken-down portions of the ergot to be infallibly recognised. The slide is first examined under a power of 100 to 120, when the powerful refracting power, the colour—deep violet on the edge, and greenish-yellow within—and the furrowed outlines of the ergot are all characteristic. A second examination with a power of 300 to 400 enables any doubtful particles to be identified.

F. H. L.

The Iodine Number of Lard at Different Times of the Year. L. van Italic. (*Apoth. Zeit.*, 1895, x., 694; through *Chem. Zeit. Rep.*, 1895, 329.)—After experiments lasting a twelvemonth, the author is unable to trace any variation in the iodine number of lard dependent on the different seasons of the year.

F. H. L.

Detection of Fluorine in Beer. J. Brand. (*Ztschr. ges. Brauw.*, 1895, xviii., 317; through *Chem. Zeit. Rep.*, 1895, 327.)—One hundred c.c. of the beer are made slightly alkaline with ammonium carbonate, then heated, and 2 or 3 c.c. of a 10 per cent. solution of calcium chloride added. After boiling for a few minutes, the liquid is filtered through a plain (not folded) filter, and the precipitate washed slightly and dried. It is introduced into a 25 c.c. platinum crucible, moistened with 1 c.c. of strong sulphuric acid, and kept for an hour at the boiling-point. The crucible is covered with a watch-glass, the convex side of which is protected from the hydrofluoric acid by a coating of wax, in which a pattern is scratched, while the concavity contains a lump of ice to keep the cover cool, with a wick syphon to remove the melting water. In this manner 1 milligramme of ammonium fluoride may be detected in 100 c.c. of beer; and by preparing a number of watch-glasses etched by

the action of increasing amounts of the fluoride (0.5 to 5 milligrammes per 100 c.c.), the process may be rendered approximately quantitative. F. H. L.

Zinc Sulphate as a Precipitant for Albumoses. A. Bömer. (*Zeit. anal. Chem.*, 1895; *Fünftes Heft.*, pp. 562-567.)—The precipitation of albumoses by saturated salt solutions and by alcohol depends on the attraction of these reagents for water. Hence, ammonium sulphate, which is soluble in cold water in the proportion of 76.8 parts per cent., is especially suitable for this purpose. The great disadvantage attending its use is the introduction of ammonia, which must be removed before the nitrogen in the precipitate can be determined. Of other readily soluble salts, zinc sulphate appeared most promising to the author, its solubility being 135 parts in 100 of cold water, and experiments were accordingly made to determine whether it could take the place of ammonium sulphate.

The precipitations were carried out in exactly the same way as in the case of the ammonium sulphate method, the precipitate being washed with a cold saturated solution of zinc sulphate. The filter and its contents were then placed in a Kjeldahl flask, and the nitrogen determined in the usual way. In each case, before adding the zinc sulphate, 1 c.c. of dilute sulphuric acid (1.4) was added to prevent the precipitation of zinc phosphate.

The results obtained with different meat extracts were as follows :

			Albumose Nitrogen determined by precipitation with	
			Ammonium sulphate.	Zinc sulphate.
Liebig's meat extract	1.17 per cent.	1.19 per cent.
Kemmerich's meat extract	1.55 "	1.52 "
" meat peptone	5.51 "	5.44 "
Cibil's meat extract	0.96 "	0.92 "

In no case could the biuret reaction be obtained in the filtrates. In the case of the zinc sulphate filtrate the test was made in two ways :

1. The saturated zinc sulphate solution was freed from zinc by adding saturated sodium carbonate solution. The filtrate was concentrated on the water-bath, made strongly alkaline with sodium hydrate, after which several drops of a 2 per cent. solution of copper sulphate were gradually added.

2. Since zinc hydrate is soluble in excess of sodium hydrate, the test was also made directly with the somewhat diluted zinc sulphate solution.

A further advantage in the use of zinc sulphate is that the peptones, flesh bases, etc., in the filtrate may be at once precipitated with phospho-tungstic acid, which is not possible in the ammonium sulphate method, since ammonia itself is precipitated by the reagent. An equal volume of dilute sulphuric acid (1.4) should be added, and then the phospho-tungstic acid.

In the four meat preparations examined the amount of nitrogen found in phospho-tungstic precipitate was :

			Nitrogen.
Liebig's meat extract	5.31 per cent.
Kemmerich's meat extract	4.05 "
" meat peptone	3.16 "
Cibil's meat extract	1.11 "

The author concludes, from these experiments, that the albumoses are completely precipitated by zinc sulphate in the case of Cibil's extract and Kemmerich's peptone. In the case of the other two, the negative results of the biuret reaction cannot be accepted as complete proof, on account of the dark colour of the solution. With regard to the behaviour of the zinc sulphate towards the ammonium salts, it would be reasonable to expect the difficultly soluble double salt— $(\text{NH}_4)_2\text{SO}_4 \cdot \text{ZnSO}_4 \cdot 6\text{H}_2\text{O}$ —to be formed; but the author found that the nitrogen usually assigned to the ammonium salts was invariably contained in the filtrate from the zinc sulphate precipitate. In the case of Kemmerich's meat extract the ammoniacal nitrogen determined directly in the original extract by distillation with an excess of magnesium oxide was 0.413 per cent., while in the filtrate from the zinc sulphate precipitate the amount found was 0.415 per cent. Hence, it is possible that the ammonia obtained by distillation with magnesia is not derived from ammonium salts, but from some other nitrogenous compounds.

C. A. M.

On the Composition of Meat Extract. J. König and A. Bomer. (*Zeit. anal. Chem.*, 1895; 5th Heft, pp. 548-562.)—While there can be no doubt that nearly all the constituents of the muscular fibre which are soluble in cold water will be found in the meat extract, the presence of gelatin or other decomposition products of the nitrogenous matter is by no means a certainty. Since the extract is prepared at low temperatures, and is only at the end concentrated to the required consistency after filtration, the amount of gelatin can only be excessively small. This view receives confirmation from the experiments of E. Beckmann (*Hilger's Forsch. üb. Lebensmittel*, 1894, p. 423), who could only find 0.5 per cent. of albumin and gelatin in Liebig's extract by precipitation with formalin. On the other hand, Kemmerich (*Zeit. f. Physiol. Chem.*, 1894, xviii., p. 409) endeavoured to prove that in South American meat extract there was about 6 per cent. of gelatin, and about 30 per cent. of albuminoids, in the form of albumoses, peptones, and other soluble compounds. In his analyses he employed fractional precipitation with alcohol of different strengths, as well as precipitation with ammonium sulphate and sodium phosphotungstate. By these means he found, in addition to flesh bases, the following amounts of albuminoids in this meat extract:

	Per cent.
1. Gelatin precipitated by 50 to 60 % alcohol	6.19
2. Albumoses precipitated by 80 % alcohol	14.76
Of these there were precipitated by $(\text{NH}_4)_2\text{SO}_4$	9.89
Other albuminoids soluble in $(\text{NH}_4)_2\text{SO}_4$	4.87
3. Peptones soluble in 80 % alcohol, precipitated by sodium phosphotungstate	12.31
	<hr/> 33.26

Since the meat extract contained 8.13 per cent. of total nitrogen, these figures gave 6.5 per cent. of this to the albuminoids, which, in the authors' opinion, was extremely improbable. They therefore critically examined the work of Kemmerich and of Stutzer (*ANALYST*, xx., 246), and by precipitation of the meat extracts with alcohol

of different strengths and determination of the nitrogen in the precipitates they obtained results considerably lower than those of Kemmerich.

For South American meat extract

		Gelatin (?) precipitated by : 50 to 60 per cent. alcohol.		Albumoses precipitated by 80 per cent. alcohol.
Kemmerich found	...	6.19 %	...	14.16 %
König and Bömer	...	1.83	...	4.50

These differences were too great to be accounted for by variation in the meat extracts, and must have been due to difference in method, Kemmerich having determined the amount of his precipitates gravimetrically, and not by direct estimation of the nitrogen.

The authors then made comparative determinations by precipitation with 80 per cent. alcohol and precipitation with ammonium sulphate, with the following results :

		Liebig's extract. Per cent.	Kemmerich's extract. Per cent.	Kemmerich's peptone. Per cent.	Cibbl's extract. Per cent.
Total nitrogen	...	9.32	8.94	9.88	2.77
Precipitated by 80 per cent. alcohol	...	0.69	1.05	4.05	0.61
Corresponding to albumoses	...	4.31	6.56	25.31	3.81
Albumoses obtained by satur- ation with ammonium sul- phate	...	7.32	9.71	34.44	5.97

These results, and the fact that in the filtrate from the 80 per cent. alcohol precipitation the biuret reaction was always obtained, showed that albuminoids were still present, and it was extremely doubtful whether these were to any extent peptones.

The usual method of determining the peptones is to precipitate with sodium phosphotungstate, determine the nitrogen in the precipitate, and deduct from this the albumose nitrogen previously determined.

In this determination the figures obtained were :

		Liebig's extract. Per cent.	Kemmerich's extract. Per cent.	Kemmerich's peptone. Per cent.	Cibbl's extract. Per cent.
Nitrogen in phosphotung- state precipitate	...	6.27	5.59	8.29	2.00
Albumose nitrogen	...	1.17	1.55	5.51	0.96
Peptone (?) nitrogen	...	5.10	4.04	2.78	1.04

It is obvious that so large a quantity of peptone nitrogen cannot be present—at any rate in the meat extracts, and that the flesh bases must claim a considerable amount of it. All flesh bases, together with the rest of the nitrogenous constituents, are precipitated by sodium phosphotungstate if they are allowed to stand for sufficient time, and therefore this reagent cannot give any idea of the amount of peptone present. Basing their conclusions largely on the absence of the biuret reaction in the filtrate of a meat extract, the authors believe that the extracts

examined contained either no peptone at all, or, at most, very slight quantities (2 to 3 per cent.).

They assigned the nitrogen found as follows :

	Liebig's extract.		Kemmerich's extract.		Kemmerich's peptone.		Cibbl's meat extract.	
	Substance.	Nitrogen.	Substance.	Nitrogen.	Substance.	Nitrogen.	Substance.	Nitrogen.
Total nitrogen	9.28	100	9.14	100	10.08	100	2.77	100
1. Soluble albumin	trace	trace	0.08	0.87	0.06	0.59	trace	trace
2. Nitrogenous compounds insoluble in 60-64 per cent. alcohol	0.21	2.26	0.33	3.61	1.36	13.49	0.25	9.02
3. Albumoses	0.96	10.34	1.21	13.24	4.15	41.17	0.70	25.27
4. Peptones	0 to trace	0 to trace	0	0	0	0	0	0
5. Flesh bases	6.81	73.38	5.97	65.32	3.97	39.38	1.56	56.31
6. Ammonia	0.47	5.06	0.41	4.49	0.29	2.88	0.09	3.25
7. Other Nitrogenous compounds	0.83	8.96	1.14	12.47	0.25	2.49	0.17	6.15

As regards the chemical examination of meat extracts, the authors remark :

1. Precipitation with 80 per cent. alcohol is of no value in determining the kind of nitrogen.

2. Albumoses should be determined by salting out with ammonium sulphate or zinc sulphate.

3. The filtrate from the ammonium or zinc sulphate precipitates should be decolorized with animal charcoal, and tested for peptones by the biuret reaction.

4. A determination of the ammonia by distilling an aqueous solution of the extract with ignited magnesia is valuable.

5. When peptone has been proved to be absent, the nitrogen in the phosphotungstate precipitate, after deducting the nitrogen derived from gelatin, albumoses, and ammonia, may be ascribed to the flesh bases. The precipitate should stand at least one day.

6. The difference between the total nitrogen and the nitrogen in the form of gelatin + albumoses + flesh bases + ammonia gives the amount of nitrogen present in compounds not precipitated by phosphotungstic acid.

C. A. M.

The Estimation of Gelatin in Meat Extracts and Commercial Peptones.

A. Stutzer. (*Zeit. anal. Chem.*, 1895; 5th Heft., pp. 568-570).—The chief difficulty in the examination of these articles is the determination of the nitrogen present in the form of gelatin. Since his last communication (see ANALYST, xx. 248), the author has found that the best process for estimating this constituent is as follows : From 5 to 7 grammes of dry, and from 20 to 25 grammes of fluid preparations are weighed into a tinfoil basin, and sufficient hot water added to dissolve the extract. Ignited sand, which has been freed from fine dust by a sieve, is then added in sufficient quantity to absorb the whole of the fluid, and the basin is placed in the water-oven until the weight becomes constant. The sand and extract are then ground in a mortar, the tinfoil cut into small strips, and the whole placed in a beaker, where it is extracted four times with 100 c.c. of absolute alcohol, the supernatant fluid being each time removed by filtration through an asbestos filter.

The residue is now treated with a mixture of alcohol and ice-water, prepared by mixing in a large flask 100 grammes of alcohol with about 300 grammes of ice, and adding sufficient distilled water to bring the total weight up to one kilogramme. This flask and four beakers (*b*, *c*, *d*, and *e*) are placed in a bath filled with broken ice. Into the beaker *a*, containing the sand, peptone, etc. (which is also placed in the ice bath), about 100 c.c. of the alcoholic ice-water are poured, care being taken that the temperature of the mixture does not exceed $+5^{\circ}$ C. After stirring with a glass rod for about two minutes, the supernatant liquid is poured into beaker *b*, a piece of ice being added at the same time. The extraction in beaker *a* is then repeated with a fresh portion of alcoholic ice-water, the liquid being decanted into beaker *c*; and this process is continued until the liquid above the sand is completely colourless. Upon each repetition the fluid is poured into a fresh beaker, and the extraction is generally complete after this has been done four times.

In order to filter the extracts, three asbestos filters are used. These consist of a funnel about 7 centimetres in diameter at the top, in which a perforated porcelain disc about 4 centimetres in diameter is placed, this being covered with long-fibred asbestos. The first filter receives the liquid in beaker *a* and the insoluble residue, with the exception of the sand. The contents of beaker *b* are poured upon the second filter, while the third filter is used for *c*, *d*, and *e*. After being well washed with the alcoholic ice-water, the whole of the asbestos filters (including the one used in the treatment with absolute alcohol) and the sand in beaker *a* are repeatedly boiled with water in a porcelain dish, the filtrate concentrated by evaporation, and the residue used for the determination of the gelatin nitrogen.

When carried out in exact accordance with these details the estimation presents no difficulties. A Bunsen's water-pump may be used to accelerate the filtration through the asbestos, but should be very gradually applied. C. A. M.

TOXICOLOGICAL ANALYSIS.

The Presence of Solanine in Potatoes. N. S. Klepzw. (*J. ochran. narod. sdruhen.*, 1895, v., 659; through *Chem. Zeit. Rep.*, 1895, 338.)—Otto's method for the extraction of solanine is very convenient where the alkaloid exists in the crystalline condition, as in the buds of germinating potatoes, but less suitable for the treatment of the rind and of the body of the tubers, where it is found in an amorphous state. In November no solanine could be detected in new potatoes after peeling; but in December, after an artificial germination, the rind contained 0.11 per cent., and the buds 0.19 per cent., while the potato itself yielded only a slight reaction. In March, potatoes which had sprouted in the ordinary manner contained in the peel 0.07, in the buds 0.2, and in the tubers 0.024 per cent. of solanine.

The author does not consider that potatoes that have germinated can be harmful as food, so long as they are properly peeled and prepared, since Clarus has found, experimenting on himself, that 0.4 gramme of solanine (corresponding to 8 kilogrammes of potatoes freed from buds) was required to produce symptoms of poisoning.

F. H. L.

The Toxicological Detection of Aqua Regia. P. Mola. (*Boll. chim. farm.*, 1895, xxxiv., 513; through *Chem. Zeit. Rep.*, 1895, 327.)—Mixtures of flesh (100 grammes), water (500 c.c.), and aqua regia (2 c.c.) do not give off, at ordinary temperatures, the smallest traces either of chlorine or of hydrochloric acid; only a little nitrous acid can be detected. Even on fractional distillation the distillates remain neutral till the temperature reaches 190° C., when nitrous acid comes over, but neither nitric nor hydrochloric acid is to be found. The best method for the examination of organic substances is that already described by Vitali for the detection of hydrochloric acid. The article to be tested is warmed in a porcelain basin to 50° or 60° C., and finely-powdered quinidine stirred in until the acid reaction disappears. The liquid is filtered, concentrated, and mixed with two-thirds of its volume of chloroform. Enough alcohol is then added to dissolve the chloroform, and finally, water to reprecipitate it. The quinidine salts thus remain in the chloroform, and on evaporation of the solvent, the acids may be detected in the ordinary manner.

F. H. L.

ORGANIC ANALYSIS.

The Determination of the Heating Effects of Coals. W. Noyes, J. McTaggart, and H. Craver. (*Journ. Amer. Chem. Soc.*, 1895, xvii., pp. 843-849.)—The work here described was undertaken in order to compare the results obtained with Hempel's calorimeter with those calculated from analyses and those obtained by Berthier's test. Six specimens of representative Indiana coal were used, the first two being non-coking, and the others coking and bituminous. The methods of obtaining the results tabulated below were as follows:

1. *Moisture*.—One gramme of coal was dried for an hour in a toluene bath (105° C.).

2. *Ash*.—The residue from the above was ignited over a Bunsen burner.

3. *Fixed Carbon*.—One gramme of coal was heated for 7 minutes in a covered platinum crucible with the full flame of a Bunsen burner. The residue, less the ash, gave the "fixed carbon."

4. *Volatile Combustible Matter*.—The loss of weight in 3 less the amount of moisture.

5. *Carbon and Hydrogen*.—About two-tenths of a gramme of coal was burned in a current of oxygen in a hard glass tube containing copper oxide and lead chromate.

6. *Nitrogen*.—Determined with soda-lime.

7. *Sulphur*.—Determined by Eschka's method, using potassium carbonate and magnesium oxide, by the method of Carius, and also by the use of sodium peroxide as follows: Half a gramme of coal was weighed into a platinum dish, 3 grammes of sodium peroxide, and a little water added, and the whole evaporated to dryness and ignited. This was repeated after adding another 2 grammes of sodium peroxide and some more water. The mass was then boiled with water, filtered, acidified, and the sulphate determined in the usual manner. The results agreed well with those obtained by Eschka's method, but were somewhat lower than those given by Carius's method.

8. *Oxygen*.—This is usually calculated by subtracting the other constituents

from 100. But where much sulphur is present an appreciable error is introduced. Assuming that the sulphur is usually present as iron pyrites, a correction was applied to the ash by adding to it five-eighths of the weight of sulphur present.

The heating effect was calculated for the coal by the formula: $8080 C + 28800 (H - \frac{1}{8}O) + 1582 Fe + 2162 S$.

Calorimetric Determinations.—Hempel's calorimeter was used (see *Zeit. angew. Chem.*, 1892, p. 393). At least three determinations were made with each coal, and a correction of 610 calories per gramme of water was made for the water condensing in the calorimeter, the average temperature of which was taken as 26° C.

Berthier's Test.—One gramme of coal was intimately mixed with 40 grammes of litharge, the mixture being placed in a Battersea C crucible, and covered with a layer of salt. The covered crucible was then placed in a hot gas furnace for 15 or 20 minutes, after which the button of lead was obtained, cleaned, and weighed. Theoretically 1 gramme of lead should equal 234 calories, but this gives results about 12 per cent. too low. In the following table an empirical factor of 268·3 calories per gramme of lead was employed, this being the average of the results obtained. The average difference of the results from the mean for a given coal was 0·11 gramme, which corresponded to 29 calories, or about 0·45 per cent.

The differences between the results of calorimetric determinations and those obtained by other methods are given in percentages:

	New Pittsburg, A.	New Pittsburg, B.	Lancaster.	Brazil.	Shelburn.	Shop.
Moisture	6·83	5·89	12·66	8·98	8·63	2·36
Volatile combustible matter	39·92	42·23	37·44	34·49	38·82	31·11
Fixed carbon	39·93	40·40	47·22	50·30	43·45	42·44
Ash	13·31	11·48	2·68	6·23	9·05	24·09
Carbon	62·88	65·26	71·41	70·50	66·86	57·32
Hydrogen	5·07	5·17	5·56	4·76	5·30	4·56
Nitrogen	1·01	1·17	1·54	1·36	1·50	1·44
Oxygen	13·06	13·25	18·42	16·29	15·69	9·93
Ash (corrected)	17·98	15·15	3·07	7·09	10·65	26·75
Sulphur	7·46	5·88	0·62	1·39	2·57	4·25
Iron (calculated)	6·53	5·14	0·54	1·22	2·25	3·72
Calories (per gramme), C ...	5081	5272	5770	5696	5402	4632
„ (calculated), H ...	991	1011	939	784	962	956
„ „ S ...	161	127	13	30	55	92
„ „ Fe ...	103	81	9	19	36	59
Total	6336	6491	6731	6529	6455	5739
Difference per cent.	+ 2·6	+ 1·2	+ 0·4	- 4·6	- 1·2	- 1·2
Calories per gramme (Berthier's test, factor 268·3)...	6307	6471	6831	6689	6461	5726
Difference per cent.	+ 2·1	+ 0·9	+ 1·9	- 2·3	- 1·1	- 1·4
Calories per gramme (calorimeter)	6175	6415	6703	6846	6532	5806

The results obtained from the analyses and by Berthier's test agreed better with each other than either did with the calorimetric results, and on the average Berthier's test, using the empirical factor, appeared to be more reliable than the results calculated from the analyses.

C. A. M.

INORGANIC ANALYSIS.

Rapid Detection of Tin. C. Deniges. (*Rev. Int. des Fals.*, viii., 98; through *Bull. de l'Assoc. belge Chim.*, 1895, iv., 132.)—This is based on the property possessed by a solution of brucine in nitric acid of becoming reddish violet in the presence of stannous salts.

The brucine solution is prepared by dissolving 0.5 gramme in 5 c.c. of nitric acid in the cold, adding 250 c.c. of water, boiling for ten or fifteen minutes, and making up the volume to 250 c.c.

A trace of the substance under examination is acidified with a little hydrochloric acid, evaporated to dryness, and the residue dissolved in several drops of water, and 1 c.c. at most of the cold solution of brucine added.

By this reaction, as little as 0.05 mg. of tin can be detected, even in the presence of salts of copper and iron.

In order to detect tin in metastannic acid, 1 c.c. of water, 0.5 c.c. of hydrochloric acid, and a fragment of pure zinc, are added to the sample. The tin is deposited on the zinc, and may then be detected as above.

C. A. M.

The Estimation of Graphite in Iron. P. W. Shimer. (*Trans. Amer. Inst. Min. Engin.*, 1895; through *Chem. Zeit. Rep.*, 1895, 273.)—The processes for the estimation of graphite in iron, which depend on the solution of the metal in hydrochloric acid, always yield higher results than those where nitric acid is the solvent. It is, however, not to the oxidizing action of the latter on the finely-divided carbon that this difference is due, but to the fact that the titanium carbide is broken up by nitric acid, while it is undissolved either by hydrochloric, hydrofluoric, or sulphuric acid, or by caustic potash. The author states that all crude irons contain titanium, the amounts varying from 0.05 to 0.40 per cent., while other carbides are usually present in minute traces.

One sample of iron, containing 3.334 per cent. of total carbon (by combustion), proved to have 3.206 per cent. of graphite, 0.128 per cent. of combined carbon, and 0.399 per cent. of titanium. By solution in hydrochloric acid, the yield of graphite was 3.327 per cent.—0.121 per cent. too much. The amount of carbon corresponding to the titanium found was 0.100 per cent. (? 0.096), and this, subtracted from the above surplus of "graphite," left 0.021 per cent. unaccounted for. Moreover, the carbon soluble in hydrochloric acid (probably combined with iron and manganese) was 0.007 per cent.; this deducted from the total combined carbon again left a difference of 0.021 per cent., which must be made up of the carbides of metals other than titanium.

It is therefore advisable, in analysing crude irons, to make determinations of the total carbon, of the carbon insoluble in hydrochloric acid, and of the graphite by means of nitric acid.

F. H. L.

The Separation of Iron from Beryllium. Elizabeth Atkinson and E. F. Smith. (*Journ. Amer. Chem. Soc.*, xvii., 1895, pp. 688, 689.)—The usual method of

separation by means of the solubility of beryllium hydroxide in ammonium carbonate does not always give satisfactory results. As a substitute for this, the authors have made use of nitroso- β -naphthol. The solutions used were: (1) A 50 per cent. acetic solution of nitroso- β -naphthol. (2) A ferric solution of which 10 c.c. contained iron, corresponding to 0.1278 gramme ferric oxide. (3) A solution of beryllium chloride of which 10 c.c. contained 0.1248 gramme.

To test the completeness of the precipitation of the iron by the reagent, 10 c.c. of the ferric solution were diluted with 200 c.c. of water, and 25 c.c. of the nitroso- β -naphthol solution added to the liquid, which was allowed to stand for twenty-four hours. The iron precipitate was then filtered, washed first with 50 per cent. acetic acid and then thoroughly with water. After drying, the iron nitroso- β -naphthol was mixed with an equal quantity of pure oxalic acid, and ignited in a weighed porcelain crucible until all the carbon had been burned off.

The results obtained were:

- (1) 0.1277 gramme of ferric oxide.
- (2) 0.1283 " "
- (3) 0.1277 " "

The amount taken was 0.1278 gramme.

Ten c.c. of the beryllium solution being treated in the same way, gave no precipitate even after standing for twenty-four hours.

When 10 c.c. each of the iron and beryllium solutions were treated in this manner, the ferric oxide found was:

- (1) 0.1277 gramme,
- (2) 0.1275 "
- (3) 0.1277 "

instead of 0.1278 gramme.

It was found that uranium salts could not be precipitated by this reagent. Nor did it precipitate solutions of cerous salts, of lanthanum ammonium nitrate, of praseodymium nitrate, of neodymium nitrate, of terbium, of erbium, or of sodium molybdate or tungstate.

With zirconium chloride there was an orange precipitate, and with ceric ammonium nitrate a bright scarlet flocculent deposit, the precipitation being, however, incomplete.

C. A. M.

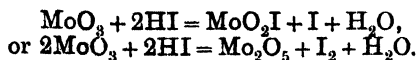
The Estimation of the Halogens in the Mixtures of their Silver Salts.
F. A. Gooch and C. Fairbanks. (*Zeits. anorgan. Chem.*, 1895, ix., 349; through *Chem. Zeit. Rep.*, 1895, 257.)—The mixtures of chloride and bromide or chloride and iodide of silver are filtered through a Gooch crucible, whose layer of asbestos is covered with a piece of platinum gauze to ensure electrical contact. The precipitate is dried at 150° C. and weighed; then melted by the direct application to it of a gas-burner, the crucible being placed on an anvil to keep it cool and prevent the melted salts soaking into the asbestos. For the separation of the chloride and bromide, the crucible is filled up with a 10 per cent. solution of oxalic acid in 25 per cent. alcohol, and treated with a current of 0.25 to 0.5 ampère for six to seven hours. This electrolyte is chosen to prevent the platinum being attacked by the nascent halogen,

but in the case of mixtures of chloride and iodide it is replaced by the following: Two volumes of 40 per cent. acetic acid are neutralized with ammonia, one volume of ammonia, one volume of 75 per cent. aldehyde, and one volume of alcohol, mixed with them. If ammonium iodate separates out on the anode, it can be dissolved by immersion in hot water; and if iodine is set free, it is a sign that the electrolyte is exhausted and needs replacing. The silver is finally washed, dried, and ignited gently in the usual manner. The latter liquid will do for the bromine separation equally well, but the former is simpler to prepare. F. H. L.

The Detection of Cyanate in Potassium Cyanide. E. A. Schneider. (*Ber.*, 1895, xxviii., 1540.)—By the employment of the azure-blue colour of cobalt cyanate, it is possible to detect from 0.35 to 1 per cent. of potassium cyanate in the cyanide. The solution to be examined must be as concentrated as possible, for on this depends the delicacy of the test. It is decomposed by a rapid current of carbon dioxide passed for about forty-five minutes, the carbonate precipitated with alcohol, and after the addition of a few drops of acetic acid, tested with cobalt acetate. In testing large amounts of the cyanide (*e.g.*, 20 grammes), they should be dissolved in the minimum of water, the bulk of the salt thrown down by alcohol, filtered, treated as before, again filtered, and a second current of carbon dioxide employed to remove the hydrocyanic acid. F. H. L.

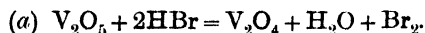
The Estimation of Molybdenum, and the Volumetric Separation of Molybdenum and Vanadium. C. Friedheim and H. Euler. (*Ber.*, 1895, xxviii., 2061 and 2067.)—I. *Gravimetric Process.*—The molybdenum is precipitated as sulphide, either by dilute acid from a solution in ammonium sulphide, or by sulphuretted hydrogen from an acid liquid. When it has thoroughly settled, it is filtered off, washed first with very dilute sulphuric acid, then with dilute alcohol, till the washings are neutral, and placed in a tared porcelain crucible, and dried at about 100° C. The crucible, with its cover on, is heated cautiously till all hydrocarbons are driven off, then uncovered, and the sulphur burnt off at as low a temperature as possible. The heat is finally raised till no more sulphurous acid is formed, the crucible allowed to become cold, the oxide dissolved in ammonia, filtered from the particles of unburnt carbon, the solution evaporated to dryness, and ignited till of a uniform yellow colour, when it is weighed as molybdenum trioxide.

II. *Volumetric Process.*—0.2 to 0.3 gramme of the molybdate and 0.5 to 0.75 gramme of potassium iodide are placed in the decomposing flask of Bunsen's apparatus, together with enough 1 to 12 hydrochloric acid to fill it two-thirds full. The heat is raised very slowly, and the liquid is only brought to the boil when the leading tube has become full of iodine vapour, and there is danger of the iodide in the condensing tubes being drawn back. The free iodine in the latter is then titrated with thiosulphate, the action taking place as follows:

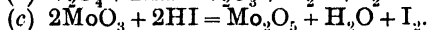
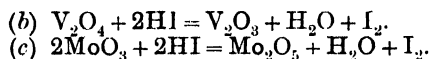


This process is available for the analysis of all molybdates, except those whose bases decompose hydriodic acid with production of free iodine, while it simplifies the examination of many complicated substances, such as the silicomolybdates and the phosphomolybdates.

III. *Separation of Molybdenum from Vanadium.*—The substance to be analysed, containing the pentoxide of vanadium and the trioxide of molybdenum, is introduced into the flask of Bunsen's apparatus as before, together with potassium bromide and fuming hydrochloric acid. The molybdic oxide is not attacked, but the vanadic oxide is decomposed according to the following equation :



The iodine liberated by the action of the bromine on the iodide in the tubes is determined as usual, and when the blue solution in the flask has cooled, 1 gramme of solid potassium iodide and 1 or 2 c.c. of syrupy phosphoric acid are added, and the distillation, etc., repeated. Both oxides are now attacked as follows :



The iodine liberated in (b) being the same in quantity as that in (a), if the amount of thiosulphate used in the first titration is subtracted from that required in the second, the remainder expresses that corresponding to the iodine in (c), or to the molybdenum present.

A number of analyses are quoted in the paper, showing satisfactory results.

F. H. L.

Estimation of Uranium in Ores which contain Phosphoric or Arsenic Acids.

R. Fresenius and E. Hintz. (*Zeit. angew. Chem.*, 1895, pp. 502, 503.)—In the ordinary methods for estimating uranium in its ores, the presence of phosphoric and arsenic acids, as well as of copper and iron, causes considerable difficulty. It is only after repeated precipitation with sulphuretted hydrogen from acid solution that it is possible to obtain the precipitate of metallic sulphides free from uranium. Moreover, the presence of phosphoric acid considerably increases the difficulty of separating iron from uranium.

Potassium ferrocyanide appeared a likely reagent to precipitate uranium in acid solution, and thus to effect a separation from phosphoric and arsenic acids; but it was found that the precipitate of uranium ferrocyanide could not be filtered off. When, however, the liquid was saturated with sodium chloride, after the addition of the potassium ferrocyanide, the precipitate rapidly settled, and could then be easily filtered off, and washed with a solution of sodium chloride. On this fact the authors have based their method of determining uranium in the special cases mentioned above.

After silica has been removed from the nitric acid, hydrochloric acid, or aqua regia solution in the ordinary way, an excess of potassium ferrocyanide is added to the dilute hydrochloric acid solution, and the liquid then saturated with sodium chloride. The precipitate containing uranium, copper, and iron ferrocyanides rapidly

settles, and is washed first by decantation and then on the filter with water containing sodium chloride, after which it is treated in the cold with dilute caustic potash. The hydrates are filtered off and washed with water containing ammonia and ammonium chloride until ferrocyanide can no longer be detected in the acidified filtrate. On treating the hydrates with hydrochloric acid they should dissolve completely. The solution of metallic chlorides is then concentrated, most of the free acid neutralized with ammonia, and an excess of ammonium carbonate added. After some time the insoluble ferric hydrate is removed by filtration, and washed with water containing ammonium carbonate.

The filtrate is heated to remove ammonium carbonate, hydrochloric acid added, and the copper precipitated with sulphuretted hydrogen, the liquid being heated at the same time.

After filtration the liquid is concentrated, the uranium is precipitated with ammonia, and the uranium hydrate converted into uranic oxide by ignition in an uncovered crucible, and weighed as such. As a control this is converted into uranous oxide by ignition in a current of hydrogen, and the amount of this also determined.

To test the method, an aqueous solution of pure uranium nitrate was used, and the following results were obtained: The amount of uranium in 25 c.c. of the solution was determined by the ordinary methods, and found to be (1) 0.2437, and (2) 0.2438 gramme.

The uranium in the solution was then precipitated with ferrocyanide, and the precipitate treated as described above: 25.250 grammes of the uranium solution yielded 0.2889 gramme of uranic oxide, corresponding to 0.2453 gramme of uranium. The amount of uranous oxide obtained was 0.2782 gramme, which corresponded to 0.2454 gramme of uranium. These figures corresponded to 0.2430 gramme of uranium in 25 c.c. of the original solution.

Control experiments with solutions containing copper, sodium phosphate, iron, and arsenic, gave equally satisfactory results.

C. A. M.

APPARATUS.

Koch's Sterilizing Cylinder as a Water-bath. A. Bau. (*Chem. Zeit.*, 1895, xix., 2003.)—The author advocates the employment of Koch's sterilizing apparatus as a water oven, especially where a number of flasks or similar vessels have to be heated exactly to 100° under precisely similar conditions. Other temperatures may be obtained by the use of liquids, having no action on metal, with different boiling points. One great advantage of the device lies in the fact that the evaporation of the heated substance is reduced to a minimum, while, as the disturbing influence of the atmospheric oxygen is in great measure obviated, the apparatus is particularly suitable for the estimation of sugar by means of Fehling's solution.

F. H. L.

An Asbestos Air-bath. S. Cerhez. (*Zeit. angew. Chem.*, 1895, p. 561.)—This consists of an asbestos basin, with a suitable cover provided with rings of different sizes, also of asbestos. The author claims that by mean of it the temperature may be easily kept constant, and an evaporation completed in one-third of the time required on a water or sand-bath. The apparatus may be obtained from F. Huger-shoff, Leipzig.

C. A. M.

REVIEWS.

GLEANINGS FROM PATENT LAWS OF ALL COUNTRIES. By W. LLOYD WISE, J.P., F.R.G.S. (London: Cassell and Co.) Price 2s.

This is a little book written by a well-known patent agent of thirty years' experience, and one which will probably be of service to chemists who may be consulted in connection with patent matters. It is practically a collection of articles, which have already been published serially, affording systematic information as to the mode of application and procedure for the grant of patent rights abroad, and for their assignment and transfer, and also as to various cognate matters. Useful statistical information is given as to the principal industries, products, imports and exports, of the countries dealt with. The series of articles is not yet concluded, so that the present volume is but the first instalment of the whole, the second part of which will be issued later. So far there are included twenty-two countries, ranging in alphabetical order from the Argentine Republic to the German Empire.

B. D.

QUANTITATIVE CHEMICAL ANALYSIS, adapted for use in the Laboratories of Colleges and Schools. (FRANK CLOWES and J. BERNARD COLEMAN.) Third edition.

PRACTICAL CHEMISTRY AND QUALITATIVE ANALYSIS. FRANK CLOWES. (J. and A. Churchill.) Sixth edition.

The rapidity with which the two books require new editions is the best proof of their excellence. It is evident that practical teaching in colleges and schools is at last departing from the "test-tubing" stage, under which it has laboured so long. Messrs. Clowes and Coleman's "Quantitative Analysis" contains an immense amount of information; and not only learners in colleges, but many who have long left college, may with profit turn to it.

The instructions for qualitative analysis are clear and complete. We note with pleasure that not only the commonly occurring elements are considered, but also the rarer ones, and that the characters of a good many organic substances are described. We shall be glad to see the list of organic substances still further extended in future editions. Since the appearance of the classical works of Fresenius, nothing nearly so good as Messrs. Clowes and Coleman's books has been placed in the hands of students.

O. H.

APPOINTMENT.

Mr. W. LINCOLNE SUTTON has been appointed Public Analyst to Norwich, *vice* W. G. Crook, deceased.

THE ANALYST.

FEBRUARY, 1896.

PROCEEDINGS OF THE 'SOCIETY OF PUBLIC ANALYSTS.

An extraordinary meeting was held on Wednesday, January 8, 1896, in the Chemical Society's Rooms, Burlington House, the President (Dr. Stevenson) occupying the chair.

The following resolution was proposed by the Chairman, and seconded by Mr. Adams: "That whereas the bye-laws of the Society state that 'The affairs of the Society shall be managed by a Council, consisting of the President, Vice-Presidents, the Treasurer, and two Honorary Secretaries, and not more than twelve other members, five to constitute a quorum,' it be added to these words that 'The Council, however, shall have power to elect as an additional member of Council the editor of the Society's journal for the time being.'"

After some discussion, the resolution was unanimously carried.

The annual general meeting was then held, the President occupying the chair. The minutes of the previous meeting were read and confirmed.

Mr. E. Russell Budden and Mr. H. Droop Richmond were appointed to act as scrutineers of the ballot-papers for the election of officers and Council for the ensuing year, and of the candidates proposed at the previous meeting for election to the Society.

The Treasurer (Mr. E. W. Voelcker) presented his report and accounts for the past year, which, on the motion of the Chairman, were unanimously adopted.

The PRESIDENT then delivered the following address:

PRESIDENT'S ANNUAL ADDRESS.

To-day the Society of Public Analysts attains its majority, for, although it is somewhat more than twenty-one years since the formation of such a society was mooted—by Messrs. Heisch and Wigner—it was not till December 1, 1874, that it was constituted. Since then we have passed through the successive stages of precarious, though strong and lusty, infancy. Vigorous childhood succeeded, with growth, and, I may add, exuberant boisterousness. Indeed, in 1877, owing to some unfortunate misunderstanding or difference of opinion connected with certain matters in the administration of the affairs of the Society, several members, including the first President, Dr. Redwood, and the first treasurer, myself, retired from the Society. It is pleasant, however, to record that Dr. Redwood, myself, and several other members, who at the time referred to felt compelled to retire from the Society, afterwards rejoined it, and took cordial part in its work. Again, we have passed through the

sedater years of youth, ever learning and feeding upon the rich stores of knowledge already provided for us. And now that the Society has arrived at full manhood, it has the promise of a long, a successful, a useful, and, I may add, an illustrious future before it. Never, however, may it be our fate to grow decrepit and useless; and should the Society ever pass away, may it only be when its work is completely and perfectly done, and when our existence shall be merged in that of some other vigorous body ready and willing to carry on the work which we now do.

A few years ago, as some of us know, hopes were entertained by some that the Society would make an honourable alliance with another Society, but the project was happily never seriously entertained by us, and our usefulness has not been impaired by our being merged in another body. Indeed, it is a matter for congratulation, and one in which we may justly take pride, that the Society has, on the whole, made steady, though not always equable, progress since the commencement of its career, and that it may now be regarded as a body of assured position, of power in public affairs, and of inestimable value and service, not only to public analysts, but to others, more especially the young and budding members of the chemical profession, supplying a need which no other society administers to. Nor do we trench upon the prerogatives of the three kindred societies—the Chemical Society, the Institute of Chemistry, and the Society of Chemical Industry.

When we look back to the earlier days of our Society, and to its first proceedings, recorded in a now scarce little volume, we see, not how modest were the aims—for they were never modest—but how small was the performance when compared with the present doings of the Society. But, although limited in amount, the earlier work of our Society was of excellent quality, and the restless energy of the first President, and the still more restless activity of the first Secretary, Mr. Wigner, must ever demand our deepest gratitude. But our means, as I well know, were small. Nevertheless, we did our best, so far as our pecuniary resources permitted.

It may be interesting for me to sketch briefly the early history of the Society, and this, as one of the founders of the Society, I am able to do, but I have to express my great indebtedness to our Secretary, Dr. Dyer, for invaluable aid.

As I have already said, Messrs. Heisch and Wigner were the chief originators of the Society of Public Analysts. A preliminary conference of Public Analysts appointed under the Act of 1872 was held at the Cannon Street Hotel on July 28, 1874. This had of course been preceded by several private meetings. On August 7 a second conference was held. Various committee meetings followed, and eventually the Society was duly constituted, and held its first ordinary meeting on December 1, 1874. The first officers and council were: President, Dr. Redwood; Vice-Presidents, Dr. Hassall and Mr. Wanklyn; Treasurer, Dr. Stevenson; Secretaries, Messrs. Heisch and Wigner; other members of Council, Messrs. Allen, Estcourt, Sutton, Bernays, Rogers and Tripe. It is gratifying to know that, after the lapse of twenty-one years, of the twelve first members of the Council six survive—just one half. The original members of the Society numbered sixty-three. Even at this early period of its existence a committee proceeded to draw up definitions of adulteration, and standards and limits for whole and skimmed milk, butter, coffee, tea and vinegar. Thus early did the Society go to the root of matters which even now greatly concern us. The Society

also got quickly to work in other directions, and on February 5, 1875, papers were read on "The Detection of Alum in Bread," by Mr. Wanklyn, "On the Analysis of Milk," by Dr. Redwood, "On the Natural Constituents of Wine," by Dr. Dupré, and "On the Decomposition of Milk," by myself—a subject still to the fore.

An arrangement was made with the proprietor of the *Chemical News*, by which our proceedings appeared in that journal, and thus the Society and its doings were brought prominently before the scientific public. Subsequently our proceedings appeared in the ANALYST, which was started by Dr. Muter in 1876, and until that journal was fully established our doings did not come before so wide a public as before, but the increase in the circulation of the ANALYST and its wider distribution, followed by the acquisition of that journal by the Society itself as its own organ, have been of inestimable benefit to us and to public analysts generally. With the new year our own organ appears in an improved form—larger and better, as we think, with more abstracts from a wider range of journals, so that the ANALYST is now incomparably the best journal of analytical chemistry published in this country—a journal of great value as recorder of advances in analytical chemistry, an excellent work of reference, and a valuable property as an asset of the Society. I think I do no more than what it is right for me to do when I express my own grateful thanks to the editor, Dr. Sykes, who makes his editorial work truly a matter of love and pleasure. I trust that our new departure, and our aim to produce a larger, better, and more useful ANALYST will prove a large success. We of the Editorial Committee hope that by inserting more abstracts from foreign journals with a wide range of foreign scientific literature, and by the more rigid exclusion from its pages of what is obviously old, or trivial, or valueless, to produce in the course of a few years a journal second to none either here or abroad as an exponent of advances in the field of analysis and technical research.

The year 1875 is memorable as the year in which the tentative Statute of 1872 was nearly entirely repealed, and the passing of the existing Act of 1875, from which the term "adulteration" disappears. With the passing of the new Statute the Society was greatly concerned. The Council was busily occupied with the consideration of the draft Bill before Parliament, and with endeavours to bring about the passing of that Bill in as satisfactory a form as possible. Official interviews with Government representatives, and non-official interviews with members of the Legislature, followed the deliberations of committees and Council, and no doubt resulted in the presence of fewer blemishes than might otherwise have existed in an Act which, though it has many defects, has proved, on the whole, excellent in working. The hard work of Lord (then Dr.) Playfair, M.P., and Sir Charles Cameron, M.P., in representing in the House of Commons the views of the public analysts of that day was acknowledged by their election as honorary members of the Society.

The Society has, as I have already said, made steady progress since its foundation, and the energy, good sense and determination of its officers and members have been abundantly proved in the past. The dissensions which unhappily arose during the earlier years of our existence were but a passing cloud. We all learn—or all who are capable of learning soon find out—that if a society is to be maintained in prosperity, honour, and public estimation, this must be done, not by glib tongues,

sharp repartee, and facility in debate, but by steady work, a kindly consideration for views and opinions which we do not altogether share, temperance in criticising others, and a keen criticism of ourselves.

Throughout the existence of the Society it has had a constant inflow of papers, nearly six hundred having been read and discussed during the twenty-one years of its working—not to speak of other papers, not read, but published in the ANALYST.

The papers read before the Society during the past year are as follows :

- "Note on the Use of Maize as an Adulterant of Oatmeal." By John White.
- "Note on a Sample of supposed Lard." By B. Kitto.
- "Note on a Sample of Lardine." By R. Bodmer.
- "On the Determination of the Bromine Absorption of Fats Gravimetrically."
By Otto Hehner.
- "On the Composition of Milk and Milk Products." By H. Droop Richmond.
- "On the Relation between Specific Gravity, Fat, and Solids-not-fat in Milk."
By H. Droop Richmond.
- "On the Maumené Test." By H. Droop Richmond.
- "Notes on Cinnamon." By Bernard Dyer, D.Sc., and J. F. H. Gilbard.
- "On the Examination of Potable Water, Chemical and Biological." By A.
Dupré, Ph.D., F.R.S.
- "The Interpretation of the Results of the Chemical and Biological Examination
of Potable Water." By J. C. Thresh, M.B., D.Sc.
- "The Composition of some English Cheeses." By W. Chattaway, T. H. Pear-
main, and C. G. Moor, M.A.
- "The Testing of Oils by the Oleorefractometer." By T. H. Pearmain.
- "The Determination and Identification of the Carbohydrates in Milk." By A.
Wynter Blyth, M.R.C.S.
- "On Formalin as a Milk Preservative." By S. Rideal, D.Sc.
- "A Comparison of the Organic Carbon and Nitrogen results obtained by Dr.
Frankland and the Companies' Analysts from the Waters supplied by the
Metropolitan Water Companies." By W. C. Young.
- "On the Use of Formalin as a Preservative of Milk Samples." By E. J. Bevan.
- "Note on Formalin." By H. Droop Richmond and L. K. Boseley.
- "On the Heat evolved on Brominating Oils." By Otto Hehner and C. A.
Mitchell, B.A.
- "Feeding Experiments with Indian Peas containing *Lathyrus Sativus*." By
John Hughes.
- "Pseudo-Woolen Fabrics." By E. G. Clayton.
- "Note on Artificial Coffee." By T. H. Pearmain and C. G. Moor, M.A.
- "Note on Adulterated Sandal Wood Oil." By T. H. Pearmain and C. G.
Moor, M.A.
- "Duclaux's Method of Estimating Volatile Acids, and the Laws of Volatility."
By H. Droop Richmond.
- "On the Presence and Proportion of Free Fatty Acid in Oil-cakes and other
Feeding-Stuffs." By Bernard Dyer, D.Sc. and J. F. H. Gilbard.

- "Note on the Bromine and Iodine Absorptions of Linseed Oil." By Rowland Williams.
- "Note on a Recent Milk Case involving an Example of Abnormal Milk." By R. Bodmer.
- "Note on 'Filled Cheese.'" By R. Bodmer.
- "The Composition of Condensed Milk." By T. H. Pearmain and C. G. Moor, M.A.
- "Note on the Composition of Commercial Condensed Milk." By Alfred H. Allen.
- "Note on the Estimation of Minute Quantities of Metals in Liquids." By E. Russell Budden and H. Hardy.
- "Note on a convenient form of Polarimeter for examining Essential Oils." By E. Russell Budden.
- "The Microscopical Examination of Water." By W. J. Dibdin.

Some of these communications are of a high order of merit, and the discussion which followed the papers of Drs. Dupré and Thresh on water examination were most interesting and valuable. The claims of chemical analysis, which had been too much aspersed by bacteriologists, were ably vindicated; whilst the value of bacteriology was duly acknowledged. A paper by Mr. Dibdin, on the microscopy of waters, formed a fitting addendum to those previous communications I have referred to. Since the discussion of last spring, bacteriology, in the hands of Dr. Klein and others, has made distinct advances in the solution of disease-problems connected with water-supply, and in the not distant future we may expect that chemical analysis and bacteriology will go hand in hand in the solution of questions arising as to the relations of water-supply to outbreaks of disease.

So far back as 1880 the Society took in hand the question of water-analysis, though, singularly enough, it is a question which little concerns us *quid* public analysts; and a Committee was appointed to consider the best methods of performing, and reporting on, water-analyses. In 1881 the report of the Committee was published in the ANALYST. The instructions and suggestions laid down in this report were most valuable, and have been adopted generally, not only by public analysts, but by a large proportion of those chemists who undertake these analyses.

Throughout the whole of our career the subject of milk-analysis, and standards and limits for milk, have been prominently before us; and it is to be regretted that no final settlement of limits has been arrived at, and conflicts between chemists and the Government chemists arise still. So far back as 1883 a committee was appointed by the Society to consider and determine the best method of milk-analysis. The committee continued its labours for nearly two years. Their report to the Society was published in 1885, and recommended the Adams process for the determination of butter-fat. It marks one of the most important points in the history of the Society, as, up to that time, each individual analyst worked by his own particular modification of some one or other's modification of milk-analysis; and as nearly all the methods in use were imperfect as regards the extraction of fat, the results of different analyses were hardly comparable in terms of one another. It was hoped that the differences between the views of public analysts and the

Government chemists were about to become minimized a year or two ago, and the pronouncement of Mr. Bannister before the Food Products Adulteration Committee that the limits of the Government chemists were at that time 8.5 per cent. for solids-not-fat, and 2.75 per cent. for fat, encouraged public analysts, since they believed that these limits would be adhered to. Our expectations have, however, not been fulfilled. Some of us, who have certified milks containing less than 2.75 per cent. fat as deficient in this constituent, have found that the Government chemists, though also finding less than 2.75 per cent. fat, have pronounced the milks to yield no evidence of deficiency. The old cry again arises that it is impossible to predict what amount of change will take place on keeping in a non-sterilized commercial milk; and still less to ascertain, with even approximate accuracy, the amount of solids-not-fat originally present in a milk which is much decomposed at the time of analysis. Every pretension to the contrary should be keenly scrutinized; and until those who make the pretension submit their methods of procedure and calculation to public criticism, they cannot expect their conclusions to be accepted as valid. Mr. Bannister has publicly stated that the new and analytical method of arriving at original solids-not-fat yields figures substantially the same as the old Somerset House time-allowance, which public analysts do not accept, and for sufficient reasons. Ere long, before a reappointed Food Products Adulteration Committee, I hope the subject will be thrashed out. I am confident that eventually our contentions, based upon sincere and deep conviction, will be accepted. Our earnest endeavour in the near future must be to secure limits below which articles of food, drink, and drugs must not be allowed to fall—limits fixed by some competent and authoritative body, on which public analysts must, of course, be adequately represented. I am not insensible to the fact that limits are but imperfect instruments; but I feel assured that they present less evils than an absence of authoritative standards, and the manner in which they have worked, *e.g.*, in the British Pharmacopœia, is highly encouraging. We must press for the establishment of standards and limits for the composition of all definite and well-recognised articles of food, drink, and drugs. Evidence as to what these standards and limits should be is daily accumulating, and our members are not slow in producing the evidence on which they should be based. The joint deliberations of public analysts and commercial men of high standing, equally interested with ourselves in securing for the public articles of fair quality, ought to suffice for the securing of just, equitable, and sufficient standards and limits.

During the forthcoming year much useful work lies before the Society, not only in the improvement of methods of analysis, but also in work of a public nature. There is little doubt that the Food Products Adulteration Committee will be re-appointed with such alterations as are necessitated by political changes, and, if so, we shall claim, and I hope obtain, a hearing. The most useful services that Mr. Hehner has rendered to public analysts by his care and vigilance in attending the meetings of the Committee, and watching over the interests of public analysts, deserve a generous recognition. We all tender him our cordial thanks for his past services in this respect, and hope and anticipate that he will continue them until our ends are achieved. Watchfulness, preparedness, patience, courage are required; and these we know will not be wanting till and when the time arrives for action.

During the past year we have lost three of our members by death: Dr. W. Morgan, Mr. Crook, and Dr. Cresson of Philadelphia. An appreciative notice of Dr. Morgan has already appeared in the ANALYST. To all who knew him he appeared to be a man of great natural ability and energy, who raised himself by his unaided efforts to a conspicuous position in South Wales, and against odds which would have weighed many a man to the earth. He to the last bravely and nobly fought the battle of life with a smiling face. His memory will be ever dear to his fellow-labourers.

The number of Honorary Members, 10, and of Ordinary Members, 210, is exactly the same as at the close of 1894, whilst the number of Associates has fallen from 31 to 26.

Mr. ALLEN, in proposing a hearty vote of thanks to the President, said he was sure that everyone present had listened to the address with very great interest. The Society was to be congratulated on having had put before it so many interesting facts, which, although probably new to many members, brought back a flood of recollections to his own mind. He would like to express his sense of the loss which had been sustained in the death of Dr. Morgan, who had been a personal friend of his own, and a chemist for whom he had the greatest respect. As the President had said, Dr. Morgan had struggled under domestic troubles of a most heart-breaking character, but his industry, perseverance, and genius had made themselves apparent to everyone who came in contact with him. The President had also referred to the late Dr. Cresson, of Philadelphia. He (Mr. Allen) was probably the only member of the Society who was personally acquainted with Dr. Cresson, who was a man of great analytical experience, and had a very large practice, holding, among other appointments, that of chemist to the Pennsylvania Railway Company. He (Mr. Allen) once visited Dr. Cresson at his laboratory, and found him to be an exceedingly genial man, and one who took a very great interest in the work of public analysts in England.

MR. HEHNER, in seconding Mr. Allen's proposal, expressed the pleasure with which he had listened to the President's address, and hoped that the younger members of the Society would be induced thereby to study the earlier history of the Society, from which useful lessons might be drawn. He asked the President's consent to allow his address to be printed in the next number of the ANALYST.

On the motion being put to the meeting by Mr. Allen, it was carried with acclamation.

The PRESIDENT (whose rising was received with loud applause) said he was very much obliged for the cordial vote that had been passed, and to his old friends, Messrs. Allen and Hehner, for the kind manner in which they had proposed it. It would give him the greatest pleasure to see his address appear in the journal. Before concluding, he had to propose a vote of thanks to the Chemical Society for the use of their rooms during the past year, which he hoped would be cordially passed. It gave dignity to the Society of Public Analysts to meet in those rooms, where so many distinguished societies met, and he felt that they ought to be very grateful for the Chemical Society's kindness.

The vote of thanks was passed with acclamation.

The scrutineers reported that the list of Officers and Council elected for 1896 was as follows :

President—T. Stevenson, M.D., F.R.C.P.

Vice-Presidents (who have filled the office of President)—M. A. Adams, F.R.C.S. ; A. H. Allen ; Sir Chas. A. Cameron, M.D., F.R.C.S. ; A. Dupré, Ph.D., F.R.S. ; Otto Hehner ; Alfred Hill, M.D., F.R.S.E. ; J. Muter, Ph.D., M.A., F.R.S.E. (Who have not filled the office of President)—A. Wynter Blyth, M.R.C.S. ; John Pattinson ; John A. Voelcker, M.A., B.Sc., Ph.D.

Treasurer—E. W. Voelcker.

Hon. Secretaries—Edward J. Bevan, Bernard Dyer, D.Sc.

Other Members of Council—Leonard Archbutt, Bertram Blount, E. Russell Budden, Charles E. Cassal, W. Chattaway, C. H. Cribb, B.Sc., W. J. Dibdin, W. W. Fisher, M.A., Sidney Harvey, B. Kitto, H. Droop Richmond, Alfred Smetham.

Mr. A. C. Chapman, F.I.C., was elected a Member, and Messrs. Norman Leonard, B.Sc., F.I.C., and Harry M. Smith Associates.

Mr. Stevenson J. C. G. Macadam, jun., F.I.C., of Edinburgh, was proposed for election as Member, and Mr. Charles Crocker, of Swansea (assistant to Messrs. Morgan, Sons, and Seyler), as Associate.

A paper was read entitled : " Note on a Series of Analyses of a Private Water Supply," by E. Russell Budden.

THE ANNUAL DINNER OF THE SOCIETY.

This took place the same evening at the Criterion, under the chairmanship of the President. Nearly seventy-five members and guests were present, amongst the latter being Mr. Hudson E. Kearley, M.P., Mr. T. H. Elliott (Secretary of the Board of Agriculture), Dr. Thorne Thorne, C.B., F.R.S. (Medical Officer of the Local Government Board), Professor Thorpe, F.R.S., Mr. A. G. Vernon Harcourt, F.R.S. (President of the Chemical Society), Mr. M. Carteighe (President of the Pharmaceutical Society), Mr. T. H. Tyrer (President of the Society of Chemical Industry), Dr. James (Secretary of the British Institute of Public Health), and Mr. G. C. Barnes (Master of the Joiners' Company).

The toast of " The Queen " having been duly honoured,

Mr. HEHNER proposed " The Houses of Parliament." He said that all members of the Society of Public Analysts had been looking forward to the imminent reform of those Acts of Parliament under which they held office. He hoped that the attention of the Legislature might not be averted from that most necessary reform by the present complications in foreign politics. England was the first country to institute and set into operation Acts directed against food-adulteration. Other countries had not only imitated England's example, but had improved upon our Food and Drugs Acts. Circumstances had altered to such an extent in the twenty years during which these Acts had been in force, and so many defects had manifested themselves in carrying them out, that they might now be considered altogether antiquated, and in urgent need of revision. He pointed out that while originally legislation against food adulteration was undertaken in consequence of the agitation of a few individuals,

at present the public, the trade in articles of food, and the officers under the Act were unanimous in asking for more stringent legislation. The good which these Acts had done was now freely acknowledged, but far greater benefit might yet be effected. A very important step had been taken by the last Parliament towards amending legislation in this direction by the appointment of the Food Products Adulteration Committee. Although that committee, from circumstances over which it had no control, had undergone a very serious amount of decomposition, he hoped that a similar committee would very soon be reappointed and would conclude its labours. He coupled with the toast the name of a prominent member of the late committee, who had devoted himself and given his great practical experience to that committee, Mr. Hudson E. Kearley, M.P.

Mr. HUDSON E. KEARLEY, in responding, said that the condition of our foreign relations at the present time would probably render it necessary for Parliament, on its reassembling, to give close attention to external, rather than internal affairs; but he sincerely hoped that the question of food and drugs legislation would not be lost sight of. He trusted that the committee would be reconstituted, and that an early opportunity would be afforded to the public analysts of laying before that body their views, which were the outcome of a long experience of the working of the present laws, as to the form that future legislation should assume in order to reach that degree of efficiency which, in the interests both of the public and of the analyst, was so much to be desired.

Dr. J. A. VOELCKER, in proposing the toast of "The Local Government Board and the Board of Agriculture," said it was the desire of the Society that public analysts throughout the country might be brought into closer connection with these two Boards, upon whom devolved the final selection of fit and proper men to fill the appointments of public analyst and district agricultural analyst. The duties attached to the making of these appointments were not only very important ones, but were often of an extremely delicate character, and he wished to take the opportunity of impressing upon the representatives of the Boards concerned the serious responsibility which rested upon them in this matter. It might at one time have been found handy to combine with the appointment of medical officer of health that of public analyst, as a matter of economy and local convenience, but at the present time the position of public analyst was one which could not be filled properly by a man who had received only a mere smattering of chemical training. It was a position which required special training, knowledge, and experience, not only in general chemistry, but in actual public analytical work. It was the duty of bodies like the Society of Public Analysts to endeavour to promote the appointment of none but properly qualified men, and it was to those Government departments which had to investigate the qualifications of candidates that they looked for co-operation and support. They wished as public analysts to see less and less of what had been put forward as indicating continual disagreement between Government departments and public analysts. That there had not been perfect harmony in the past had not been invariably the fault of the Government departments, but had often been due to insufficiencies and incapacities upon the other side; but he ventured to think that the Society now combined in its ranks men who were worthy representatives of analytical chemistry,

and who could stand before the general public as fully qualified to undertake the duties of their position.

He would like to refer to a singular anomaly which he thought ought to be removed. Those who were district agricultural analysts under the Fertilizers and Feeding Stuffs Act were in the happy position that the chief agricultural analyst was also an official of the Board of Agriculture. From this it followed that if, as a district analyst, he (Dr. Voelcker) had perhaps a slight difference of opinion with the chief analyst on any particular point, their relative positions were such that they could both meet and talk over the matter with Mr. Elliott, and he had not the slightest doubt that, in Mr. Elliott's comfortable room, they would speedily settle all their differences without being driven to air them in a court of law for public benefit. There was a distinct advantage in having both the analyst and the official referee in the same department. He ventured, however, to put forward, by way of contrast, what took place under the Food and Drugs Act. Supposing that he was a public analyst, and that, a prosecution having resulted upon his certificate, the remaining sample was submitted to the official referee. There was no comfortable room like that of Mr. Elliott's in which possible difficulties might be settled; on the contrary, there was absolutely no official common ground upon which the public analyst and the official referee could meet, and the consequence was that the matter had to be brought into court, and an unseemly wrangle ensued between those who wished to support the public analyst and those whose object was the reverse. This was not an edifying spectacle either for analysts or for the public, and it seemed to him that it would be a very good thing if it could be removed. They, as analysts, ought to be able to discuss with the officials points on which opinions might be at variance, and thus "agree with thine adversary" before going into court, and not, as at present, be compelled to bring such matters into public review, and before magistrates who knew little or nothing about the real points involved.

On the part of public analysts generally there was a feeling that something like a committee should be formed, on which public analysts would be fully represented, and in this way they might discuss matters with the officials of the Inland Revenue and other departments concerned. In this way there might be some understanding come to as to what "limits" should be fixed, so that they might not go into court as a set of scientific men who could not agree one with the other. The public thought more of the disagreements between them than of the science which they professed to practise.

The Board of Agriculture was much to be congratulated for the efforts which it had made towards suppressing the importation of foreign adulterated butter. He was sure that public analysts would find in Mr. Elliott a sympathetic officer, and one, moreover, who was willing to act on behalf of the public good. At the same time, it was satisfactory to note that under Dr. Thorpe's rule a point had been reached when samples that really ought not to "pass" would no longer be allowed to do so, on the ground of their being up to the quality of some exceptional and abnormal sample that had at some time or other been discovered. As public analysts, they might now begin to feel satisfaction that the "standards" adopted by the Society of Public Analysts were being more and more nearly reached by the official referees and understood by the public.

Dr. THORNE THORNE, C.B., F.R.S., returned thanks as the representative of the Local Government Board, and assured the company that the aim at Whitehall was the same as the aim of the Society of Public Analysts, viz., to raise the standard and status of the public analyst in the estimation of the public and from the scientific point of view. He thought that some of the improvements alluded to by Dr. Voelcker were due to the Local Government Board. A public analyst should be, to quote Lord Macaulay, "a man of parts," and they asked in a candidate for this position, first and foremost, that he should be an expert in chemistry; second, that he should, from the nature of his avocations, be able to spend such time in his laboratory as to maintain his expertness; and next, that, since the question was one of Foods and Drugs, he should have certain qualifications in physiology, therapeutics, and microscopy. It would be well, he thought, if all candidates were required to pass a definite examination, but Parliament had seen fit to decide otherwise, and had imposed upon the Local Government Board the task of deciding as to competency. For this purpose the Board were dependent on documentary evidence, which they always required; but if there was one point upon which they had difficulty in obtaining satisfactory documentary evidence, it was as to competence in chemistry. Now, what was the remedy? When they received testimonials and certificates signed by men whose names would carry weight anywhere, they could not question their *bonâ fides*, they could not do otherwise than accept these as proofs of competency; and yet everyone knew what testimonials sometimes were. There was a gentleman he knew in another branch of life who always gave each applicant a testimonial in these terms: "If Providence preserves his life, he will be an ornament to his profession" (laughter). After he had done this for a few years, no one went to him for a testimonial (renewed laughter). In the case of a candidate for the post of public analyst who was a qualified medical man, his proficiency in physiology, therapeutics, and microscopy were amply guaranteed, and, therefore, he (Dr. Thorne) maintained that if a medical man was really an expert chemist, and had a district which would enable him to spend a large amount of time in his laboratory, he was likely to be the best public analyst; but chemistry must necessarily be regarded as the most important qualification. As he had said, the main object of the Local Government Board was to raise the status of the public analyst, to ensure as far as they possibly could that every man appointed should be competent. They quite agreed with Dr. Voelcker that the Medical Officer of Health ought not, in virtue of his office, to be public analyst. His honest opinion would be in this direction, that no Medical Officer of Health, however competent, should hold the post, if he had a district which, by reason of its population and area, was such that he could not, without neglect of his medical officer's duties, spend ample time in laboratory work. Under present circumstances, and in the absence of an examination (which possibly might be provided by the Institute of Chemistry), they were doing their very best. On the one hand, however, they had the candidates for appointments, and the local authorities—who resented the interference of the Board in the direction of demanding fuller proof of competency; and, on the other, the difficulty which the Society had often realized, of securing, as the result of mere documents, proof of real expert competency in chemistry. Under these circumstances, they could not please all men.

He had been more free, perhaps, in expressing his opinion than was customary in an after-dinner speech, but he did not speak so much as an official as one whom the Society had kindly honoured as their guest.

Mr. T. H. ELLIOTT, in responding for the Board of Agriculture, said that while Local Government was a prosperous institution, Agriculture was but a poor sister, who for the last fifteen years had been suffering from very severe depression. The Board of Agriculture were, therefore, especially grateful for the manner in which this toast had been proposed and responded to. The Society of Public Analysts had rendered valuable assistance to the Board in connection with the sale of fertilizers and feeding stuffs. The Act which regulated the sale of these articles was recent, and it was too soon to see definitely how it would work; but he thought there were signs that some assistance had been afforded to the farmer. The subject of the adulteration of fertilizers and feeding stuffs had formerly been the constant subject of discussion at agricultural meetings, but it was now but rarely mentioned. He attributed this absence of complaint to the existence of a thoroughly competent body of agricultural analysts, and he wished to express his gratitude, on behalf of the Board of Agriculture, for the able assistance which the Society had rendered in connection with the passing of the Act, and in bringing it into operation. It had been the most sincere desire of the Board that only men of the first rank should be selected to act as District Agricultural Analysts. No pains had been spared to impress upon local authorities the views of the Board as to the requisite qualifications, and the circular issued for that purpose had been framed in consultation with several gentlemen who were present before him. He would also wish to refer to the fact that a strong feeling existed that the agricultural industry would be much assisted by further measures to prevent adulteration. The work which the Board had recently undertaken in connection with the analysis of butter had led him to the conclusion that if the traders, great and small, of this country would make proper use of the science of analytical chemistry, the result would be that the evil of adulteration would practically cease to exist. It was probably necessary that further action should be taken by Parliament, but he would venture to hope that the traders and importers of this country would themselves assist by taking advantage to a greater extent of the services of analysts.

The PRESIDENT proposed the toast of "The Learned Societies." Most of those present belonged to some one or other of these societies, and they had with them the President of the Chemical Society, with which he had himself been connected for over thirty years; and whatever differences in views might exist as to the relative merits of different branches of chemical knowledge, he felt sure that they would all appreciate the services rendered to science by Mr. Vernon Harcourt and the Chemical Society.

Mr. A. G. VERNON HARCOURT, in replying, said that the claims of the learned societies to an expression of goodwill on such an occasion as the present were well understood. It was upon the researches of those who made it the whole or a part of their work to pursue knowledge for its own sake that the practical applications of science were based. The learned societies helped to maintain that internationa

character which science, perhaps, even more than literature, possessed, and which should have some friendly influence upon the solution of the grave political questions which had suddenly sprung up between us and our American and German friends.

Mr. MICHAEL CARTEIGHE (President of the Pharmaceutical Society), in proposing "The Society of Public Analysts," said that at the time when the question of adulteration was first raised in this country, it was impossible to point to a single person who possessed the necessary qualifications of a Public Analyst. Almost simultaneously with the passing of that Act which enacted that competent men should be appointed by competent authorities to stand between the public and the dishonest trader, the Society of Public Analysts was formed. The Society immediately set to work to technically educate its members up to the proper standard of efficiency, and they were to be congratulated upon the results achieved. They had encouraged every member of their body to devote his ability to some branch of research connected with public analysis, and had founded a journal whose numerous volumes contained a mass of information which was a credit to the country. They had lately been instrumental in getting Parliament to appoint a Select Committee to investigate the whole question of the sale of food and drugs, and for the assistance of that committee had drafted a Bill embodying the views which their long and detailed experience had suggested to them. He believed that the Society, if wisely directed (as it had been for many years), and loyally supported by its members, would continue to grow and prosper. Their present President, whose name he had to couple with the toast, had been associated with the Society from its commencement, and it was interesting to note that he was their first Treasurer. He was the successor to a long line of distinguished Presidents, commencing with Dr. Redwood (who might be said to have been the first Public Analyst), and who was followed by Dr. Dupré, Dr. Muter, Mr. Heisch, Mr. Wigner, Dr. Alfred Hill (of Birmingham), Mr. Allen (of Sheffield), Dr. Adams (of Maidstone), Mr. Hehner, Sir Charles A. Cameron (of Dublin), and, lastly, Dr. Stevenson. Among all these men there was not one who had not made a name in science before he became a public analyst, and most of them had contributed a distinct quatum to the advance of technical work. Dr. Stevenson was a very distinguished man in many ways; his ability and sound judgment were held in great respect by those members of the Government service with whom his work brought him into contact, and he merited the confidence of all who were connected with the sale of articles of food and drugs.

The PRESIDENT, in responding, said that public analysis had been a thing of growth. They were precipitated somewhat rashly upon the public, but he thought that they had justified their position, and that they had done as much as any other body to advance not only technical analysis, but scientific analysis also, besides obtaining a by no means unimportant voice in public affairs. Their journal undoubtedly occupied the first position among those devoted to analytical chemistry in this country, and in its enlarged and improved form would compare favourably with those of foreign countries.

Mr. CASSAL, in proposing the toast of "The Visitors," coupled with the name of Professor Thorpe, said that it afforded a great deal of pleasure to the members of the

Society to welcome so many distinguished guests upon that occasion. Their presence was a sufficient guarantee of the high appreciation in which the Society's work was now held by those who, from various points of view, might properly be regarded as amongst the best qualified to judge of it. The presence of Dr. Thorpe was especially gratifying to the Society. As public analysts, they could not wish for a man possessing better qualifications to enter upon the direction of the enormously important Government Department of which Dr. Thorpe was now the head. He believed that he spoke with the full approval of his colleagues when he said that Dr. Thorpe would assuredly command the respect and confidence of the public analysts of Great Britain in the difficult and delicate post which he had accepted. There had, of course, been various differences of opinion in the past between public analysts and the chemists of Somerset House, but those differences, he fully hoped, belonged to the past. Should any even serious differences or disagreements as to the analytical data and the value of analytical processes occur in the future, he believed they would be easy of adjustment, for he was convinced that they could be settled by amicable and full and free discussion in the true scientific spirit. He trusted and believed that in the future, far from there being differences of opinion and divergence of action between public analysts and the Somerset House Analytical Department, there would be united action directed to a common end—a condition of things which could not fail to accelerate the coming of the time when the profession of analytical chemistry would, by common consent, be held to have deserved well of the people, and would accordingly receive its just meed of recognition and respect from the nation.

Professor THORPE said that the duty which had been placed upon him was, perhaps, the most pleasurable that could be undertaken by any speaker that evening. A succession of very able speeches had been delivered by gentlemen representing the various interests and associations with which the Society was connected, but he thought that previous speakers might well envy him his present task, for of all the duties that might be imposed upon an after-dinner speaker, undoubtedly the most easy, the most pleasant, and the most grateful was that of tendering thanks to one's hosts. He and his fellow visitors who had been the recipients of the kind hospitality of the Society would leave that room with a pleasant recollection of a pleasurable evening.

As regards himself, he wished to thank Mr. Cassal for the kindly terms and gracious manner in which he had referred to him in connection with the position which it was his lot now to fill. Those present would not misunderstand him when he said that he had entered that room, he would not say in trepidation, but with a feeling of slight uncertainty as to what was going to befall him. The warmth of his reception had, however, dispelled at once even the slightest shadow of a shade of any doubt he might have entertained on that head, and he could only thank them sincerely for the kindness, courtesy, and hospitality with which he had been greeted. When he was requested to speak that evening, the saying at once occurred to him that there comes to every man golden opportunities for holding his tongue. But, however officially reticent he might have desired to be, his tongue was loosened by the kind expressions which had fallen from Mr. Cassal. He was sure it was the desire of the Society that the great body of public analysts might be able to work in

harmony with the laboratory which he had the honour to direct, and which, by an act not of their own seeking, was made, in a measure, a court of reference and appeal. If that was their sentiment, he sympathized with it from the bottom of his heart; and he had no stronger desire, in dealing with the many duties of his office, than that both he and they, public servants as they were, should work together cordially and harmoniously in the discharge of their public duties. Their end was the same—the welfare of the community—and that end could be most quickly and most certainly assured by friendly and harmonious co-operation.

Dr. Voelcker, in the course of his admirable speech, had referred in feeling terms to the unhappy fact that he (Dr. Thorpe) had not a pleasant room in which the questions which might arise from time to time between the Government Laboratory and the public analysts might be discussed in the same agreeable manner as at the Board of Agriculture, and amidst the same pleasant surroundings as it was the good fortune of his friend, the Secretary of the Board, to possess. None regretted that circumstance more keenly than himself. The moral of Dr. Voelcker's remark—and he commended it to the notice of her Majesty's Office of Works—was that the amenities of official intercourse are greatly sweetened by pleasant surroundings in a pleasant room. That fact, he was afraid, was too frequently lost sight of in the public service. But he might say that, thanks to the beneficent act of the Treasury, he was shortly to be placed in possession of a building which, he thought, would, in its fittings and appointments, do more justice to, and be more worthy of, the science which they professed. He could not as yet say what his office would be like, but, at all events, he hoped that it would not be unworthy of a visit from them; and he trusted that its surroundings and comforts would be such that he might look forward to that personal adjustment of such questions as had been referred to, and that he might enjoy the same pleasant facilities for such adjustment as were possessed by Mr. Elliott. He concluded by again expressing his hearty thanks for the manner in which the toast had been proposed and received (loud applause).

Mr. T. H. TYLER (President of the Society of Chemical Industry) proposed "The Editorial Committee" of the ANALYST. The responsibility attached to the carrying on of such an organ as the ANALYST was a very weighty one, and the manner in which that responsibility was borne by those on whom it rested was worthy of high compliment.

In the absence, through illness, of Dr. Sykes, Mr. ALLEN returned thanks on behalf of the Editorial Committee.

Mr. CARTEIGHE proposed the health of "The Honorary Secretaries," expressing a sense of hearty appreciation of their labours on behalf of the visitors.

Dr. BERNARD DYER returned thanks, and the proceedings came to a close.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD ANALYSIS.

Estimation of Tannin in Wines. E. Manceau. (*Compt. Rend.*, 1895, cxxi, 646; through *Chem. Zeit. Rep.*, 1895, 372.)—One hundred c.c. of the wine are put into a stoppered flask, 1 gramme of catgut added, and allowed to stand for a week at 15° C. Twenty-five c.c. are then titrated with a permanganate solution of which 1 c.c. is equal to 0.2 milligramme of gallo-tannic acid, using indigo-carmin as an indicator. A similar titration is carried out on the original sample, the amount of tannin being obtained from the difference.

For the catgut the author recommends the employment of uncoiled violin-strings, which have been washed in dilute alcohol, acid, and water, until they have no further action on permanganate in the cold. F. H. L.

Determination of Fat in Milk. H. Tiemann. (*Milch Zeit.*, 1895, xxiv., 714; through *Chem. Zeit. Rep.*, 1895, 372.)—From results obtained at the "Milchwirtschaftliche Versuchsstation" at Kiel, the author concludes that it is not necessary to adopt any method for the sterilization of samples of milk when Wollny's process for the estimation of the fat is employed. None of the micro-organisms which cause the putrefaction of milk, etc., appear to affect the fat in any way. F. H. L.

The Variation in Size of the Fat-globules in Cows' Milk. E. Gutzeit. (*Landw. Jahrb.*, 1895, xxiv., 539; through *Chem. Zeit. Rep.*, 1895, 372.)—The average size of the fat-globules in the milk of any cow is affected by a number of external circumstances. Any sudden change in the weather, the food, or the living-place, may produce considerable alteration, although it is impossible to assign any uniform variation, i.e., any regular increase or decrease in size of all the globules, to any one of these causes. During the whole time of lactation they regularly diminish in diameter. Among cows of the same breed the average size of the particles extending over the whole lactiferous period is fairly constant, and in the milk of each particular individual the globules, whether large or small, possess the same physical and chemical characteristics. F. H. L.

Extraction of the Fat of Cheese for the Purpose of Examination. O. Henzold. (*Milch Zeit.*, 1895, xxiv., 729; through *Chem. Zeit. Rep.*, 1895, 372.)—Three hundred grammes of the powdered cheese are agitated in a wide-necked flask with 700 c.c. of 5 per cent. caustic potash, previously warmed to 20° C. After about 10 minutes the cheese dissolves, and the fat floats to the surface, where, by cautious shaking, it may be made to agglomerate in lumps. The liquid is diluted, the fat removed with a spoon, washed in very cold water, then kneaded till as dry as possible, and finally melted and filtered.

Experiments show that under this treatment the fat suffers no change in composition. F. H. L.

Artificial Colouration of Wine. **M. Rinzaud.** (*Union Pharmaceutique*, xxxvi., 446; through *Bull. de l'Ass. belge Chim.*, 1895, 231.)—For the detection of artificial colouring matter in wines the author uses sodium peroxide. This acts as an oxidizing agent, completely destroying the colouring matter of a natural wine. Wines coloured by fuchsine, etc., are also decolourized by the reagent, but the colour reappears on the addition of an acid. In making the test 5 c.c. of the wine are treated with 0.10 to 0.15 gramme of sodium peroxide. After the lapse of five minutes, when all colour should have disappeared, several drops of acetic acid are added to acid reaction.

C. A. M.

Emulsified Lard. **A. Goske.** (*Chem. Zeit.*, 1896, xx, 21.)—In a recent lawsuit in Düsseldorf it was announced that the purification of lard by means of a current of air raised its iodine number, increased its reaction with Welsman's test, and caused it to be slightly reduced by Becchi's reagent. The author has examined four samples of lard of known purity, before and after the air-treatment, and is unable to bear out these statements. One sample gave a higher iodine number, one a lower, the others practically identical values. Welsman's test gave the same results all through. Two samples gave a very faint brown colour with Becchi's test, in no way resembling cotton-oil, while the others showed no sign of reduction. He also finds that samples of pure lard, which, like these two, give a slight reaction with Becchi's reagent, if washed slightly in warm water, no longer yield the least trace of colour on the application of the test.

F. H. L.

ORGANIC ANALYSIS.

Trimethylene Glycol as a bye-product in the Glycerin Manufacture.

A. Noyes and W. H. W. Watkins. (*Jour. Amer. Chem. Soc.*, xvii., 1895, 890-891.)—During last winter a firm of soap-makers in Boston found a difficulty in getting their glycerin to the required commercial gravity. A quantity of the "light stuff" was sent to the authors for examination, and by fractional distillation they obtained from it about 38 per cent. of a liquid boiling between 214° and 217° C, and having a specific gravity of 1.056 at $\frac{20^\circ}{15^\circ}$ C. On analysis the formula of this substance was found to be $C_3H_8O_2$. It was therefore trimethylene glycol, which has a boiling-point of 214° C. and a specific gravity of 1.0526 at $\frac{18^\circ}{0^\circ}$ C. The isomeric propylene glycol boils at 188°-189° C., and has a specific gravity of 1.0403 at $\frac{19.4^\circ}{0^\circ}$.

Freund (*Monat. Chem.*, ii., 638) showed that trimethylene glycol was one of the principal fermentation products of glycerin, and the authors considered it highly probable that the glycol had been thus produced. It was probably present in the original fat, which consisted largely of house refuse fat, having owed its origin to spontaneous saponification and subsequent fermentation of the glycerin.

Its presence in glycerin used for making nitro-glycerin might be dangerous, since

it reacts with nitric acid with explosive violence. It would be detected by the glycerin giving a low specific gravity accompanied by a high result by the bichromate titration.

C. A. M.

Examination of Linseed-oil, Varnish, and Oil-colours. R. Hefelmann and P. Mann. (*Pharm. Central-H. N. F.*, 1895, xvi., 685; through *Chem. Zeit. Rep.*, 1895, 388.)—The authors advise the use of the butter-refractometer for testing these substances, stating that even small additions of rosin, rosin-oil, metallic resins, and mineral oil may be recognised by the increased rotation values observed. A number of examples are quoted.

F. H. L.

Estimation of Sulphur in Organic Compounds. A. von Asboth. (*Chem. Zeit.*, 1895, xix., 2040.)—One gramme of the powdered substance is mixed in a nickel crucible with 10 grammes of dry sodium carbonate and 5 grammes of sodium peroxide. The crucible is heated very gently until the mass falls together and begins to melt; the heat is then raised until it becomes perfectly liquid. After cooling, the melt is dissolved in water, filtered, hydrochloric acid containing bromine added, and the liquid boiled till all odour has disappeared. The sulphates are then precipitated with barium chloride as usual.

It is necessary to adhere to the proportions given for the sodium carbonate and peroxide, and to heat the crucible very cautiously at first, in order to avoid loss by spurting. Liquids and extracts may be analysed equally well by this process, after being subjected to evaporation, usually with half of the carbonate, in the crucible itself. When the remainder of the carbonate and the peroxide are added, and stirred in with a platinum wire, a very energetic reaction takes place, but with care no loss need occur. The whole test occupies only about 2 to 2½ hours in the case of solid substances, and the results quoted for potassium thiocyanate are satisfactory.

F. H. L.

Estimation of Grape or Invert Sugar in Solutions containing Lead. Zamaron. (*Bull. Ass. Chim.*, 1895, xiii., 346; through *Chem. Zeit. Rep.*, 1895, 348.)—The author finds that the best method of removing lead from liquids in which sugars are to be determined by means of copper solutions is by the aid of ammonium oxalate. The excess of ammonia is without influence on the opacity.

F. H. L.

The Reactions of Digitalin. C. C. Keller. (*Ber. Pharm. Ges.*, 1895, v., 275; through *Chem. Zeit. Rep.*, 1895, 349.)—Commercial specimens of digitalin contain four medicinally active principles: *digitonin*, a glucoside which can be split up into digitogenin, dextrose, and galactose; *digitalin*, an amorphous glucoside consisting of digitaligenin, grape-sugar, and digitalose; *digitalein*, a crystallizable glucoside not yet investigated; and *digitoxin*, a bitter substance which is not a glucoside. These may be identified by dissolving in glacial acetic acid, adding a drop of ferric chloride so dilute that the solution is only faintly yellowed, and floating it over an equal

volume of strong sulphuric acid, when colours are produced at the junction of the two liquids, which are very sharp and characteristic. Digitonin yields a faint rose, rapidly bleaching; digitalin, a permanent carmine red, having a violet cast even in the presence of .05 milligramme per c.c.; digitalein, an intense red, somewhat like the last, but less permanent. With digitoxin, a greenish-brown is first produced, changing until the acid becomes a brownish-red, while above it appears a bluish-green band, rapidly altering to indigo-blue in colour. This last reaction is also readily obtained on examining the extract or the infusion of digitalis.

F. H. L.

Valuation of Extract of Cinchona. Van L. Hulsebosch. (*Ber. Pharm. Ges.*, 1895, v., 286; through *Chem. Zeit. Rep.*, 1895, 349.)—To determine the alkaloids, 1 gramme of the extract is moistened with 5 c.c. of water and 5 drops of normal hydrochloric acid in a small beaker, introduced into the percolator, and the beaker rinsed twice with 5 c.c. of water. Ether is added and allowed to act for an hour, in order to remove all soluble matter. A tared flask is then attached, 2 c.c. of normal soda and 3 c.c. of water poured on to the substance, fresh ether added, and, after two hours, evaporated off, the alkaloids dried at 100° C. and weighed.

To estimate the cincho-tannates, 10 grammes of the fluid extract are mixed with 10 c.c. of a 20 per cent. solution of sodium acetate in a weighed glass basin with vertical sides. It is heated on the water-bath till the salts have agglomerated and attached themselves to the walls of the vessel. The liquid is then poured off, the basin rinsed with a little water, the moisture removed with a strip of filter-paper, and the residue dried at 100° C. and weighed. The weight of the tannates should represent at least 12 per cent. of the extract, and they should form, when ground up, a pale flesh-coloured powder.

F. H. L.

Estimation of Unsaponifiable Matter in Wool-fat. W. Herbig. (*Ding. Polytech. Jour.*, 1895, ccxcvii., pp. 135-141.)—The methods for this estimation are classified by Benedikt into three groups: (a) Direct saponification and subsequent weighing of the unsaponified matter, (b) Extraction of the soap solution with petroleum spirit or ether, (c) Extraction of the dry soap with these solvents. Of these processes the first invariably gives too low results, and in the author's opinion all have defects which limit their applicability and influence the accuracy of the results.

In his experiments to find a reliable method, the author tried the influence of solvents on the salts of the fatty acids obtained by precipitating a solution of the potash soap with the chlorides of copper, zinc, cadmium, aluminium, barium, strontium, calcium and magnesium. In each case they were soluble to a considerable extent in ether and petroleum spirit. The calcium salt was the least soluble, but satisfactory results could not be obtained. This difficulty was overcome by using pure acetone as the solvent, it being found that the calcium salts were completely insoluble in it. On this property the following process is based: From one to 2.5 grammes of the wool-fat are saponified with seminormal alcoholic potash, the excess

of alkali neutralized with standard acid, and the whole washed into a beaker with boiling alcohol. The alcohol is evaporated, the solution heated to about 70° to 75° C., and the fatty acids precipitated with calcium chloride, the amount of which has been calculated from the saponification equivalent. The precipitate is filtered off, well washed with dilute alcohol (1 : 20), and dried on blotting-paper *in vacuo*. When dry, it is extracted in a Soxhlet extractor with freshly-distilled acetone for six hours, after which the acetone is evaporated, the extract washed with ether into a platinum basin, and the ether evaporated, and the residue, which consists of the unsaponifiable matter, and of the esters, which cannot be saponified by the ordinary process of boiling with alcoholic potash, dried at 105° C. and weighed.

The chief points to be observed are the absolute purity of the acetone, the fraction boiling between 55·5° and 56·5° C. being used, and the temperature at which the calcium salts are precipitated. If too hot they fuse, and if too cold they become slimy, subsequent filtration being almost impossible in either case. It is also advisable to extract the cork of the extraction apparatus with ether, alcohol and acetone; the errors introduced by the hot alcohol dissolving some of the calcium salts, and by the decomposition of the latter during filtration, are so very trifling that they may be neglected.

The following results were obtained in this way with three samples of wool-fat. Five determinations were made in each case, but only the highest and lowest of these are given here :

Wool-fat.	Fat used.	Extract found.	Ash in Extract.	Per cent. unsaponifiable matter.
	Grammes.	Grammes.	Grammes.	
South American	2·1156	1·0261	0·0002	48·5
"	2·1110	1·0200	0·0001	48·31
New Zealand	2·2145	1·0283	0·0001	46·44
"	2·4928	1·1510	0·0000	46·17
Russian	2·3340	0·8704	0·0000	37·29
"	2·1526	0·7981	0·0002	37·07

C. A. M.

INORGANIC ANALYSIS.

Method for Distinguishing between Photographic Prints Produced by Development and by Direct Exposure. G. F. Jaubert. (*Monit. Scient.*, 1895, xlv., 813.)—It is stated that an ordinary fixing bath of (about) 10 per cent. thio-sulphate containing 0·2 per cent. of ammonium bichromate will not dissolve the image of a chloride-emulsion paper if it has been "developed," but will remove it if it has been obtained by insolation alone.

F. H. L.

The Volumetric Determination of Lead. A. S. Cushman and J. Hayes-Campbell. (*Journ. Amer. Chem. Soc.*, xvii., 1895, pp. 901-904.)—Among volumetric methods for the determination of lead that of Schwartz is in very general use. In

this the lead is precipitated as chromate in a sodium acetate solution, standard potassium bichromate being added until a faint red tint appears when a drop of the liquid is brought into contact with neutral silver nitrate on a porcelain plate. The objection to this is that the end point is frequently overrun, owing to the uncertainty introduced by the yellow of the precipitated lead chromate. A similar difficulty occurs in Diehle's modification of Schwartz's method (*Zeit. anal. Chem.*, 1880, 806). To obviate this the authors propose the following modification: One gramme of the pulverized ore is digested in an evaporating dish with 15 c.c. of a mixture of nitric and sulphuric acids (2 : 1) until completely decomposed. Ten c.c. more of sulphuric acid are now added, and the liquid evaporated until it fumes freely. After cooling, 10 c.c. of dilute sulphuric acid (1 : 10) are added, and then gradually 40 c.c. of water. The liquid is boiled and filtered, and the residue washed by decantation with the dilute sulphuric acid, as little lead sulphate as possible being allowed to get on to the filter. Twenty c.c. of strong ammonia are then poured upon the residue, after which acetic acid is added to slightly acid reaction. When the lead sulphate is dissolved the liquid is poured through the filter, the paper having been previously moistened with ammonia. The filter is washed with water containing ammonium acetate, and finally with hot water. The filtrate is cooled, excess of standard bichromate added from a burette, the precipitate allowed to settle, filtered off, and the filtrate titrated with standard ferrous ammonium sulphate.

The method can be carried out in about thirty minutes, and where the ore is known to be free from bismuth and antimony, the lead can be brought into solution by nitric acid alone, and immediately titrated with bichromate and ferrous ammonium sulphate.

To test the method, a sample of galena which by gravimetric methods was found to contain 86.0 per cent. of lead was treated as described above. The amount of lead obtained in eight determinations varied from 85.72 to 86.08 per cent., the mean being 85.88 per cent., so that in general it may be said that the results are slightly low.

C. A. M.

The Estimation of Sulphur and Chlorine by Means of Sodium Peroxide.

A. Edinger. (*Zeit. anal. Chem.*, 1895, pp. 362-368.)—In estimating sulphur in derivatives of the aromatic amines by the Carius method and by ignition with soda and potassium nitrate, the author found that the resulting barium sulphate required to be purified from barium nitrate. To obviate this experiments were made to determine how far barium and sodium peroxides could be relied upon as oxidizing agents.

With inorganic sulphides very good results were obtained. By treating dried copper sulphide in a platinum crucible with barium peroxide and some soda, the amount of sulphur found was 33.7 per cent., as compared with 33.60 per cent. calculated. There was, however, the drawback that the barium peroxide attacked the platinum, and the same objection applied with still more force to the use of sodium peroxide. In the latter case a porcelain crucible could be used, and any silicate removed by filtering the warm acidified sodium sulphate solution before precipitating the sulphuric acid.

Cadmium sulphide oxidized with sodium peroxide gave 22.20 per cent. of sulphur (theory, 22.10), and in copper sulphide 33.29 per cent. was found, as against 33.6 per cent. calculated. A further experiment with cadmium sulphide in a graphite crucible gave too high a result (22.7).

As a test for the simultaneous estimation of sulphur and chlorine, a mixture was prepared containing calcium sulphide, calcium chloride, and 20.2 per cent. of iron oxide and silica. This was gently warmed over a Bunsen flame with ten times its weight of sodium peroxide and soda. The mass was then treated with quite dilute nitric acid, the insoluble residue filtered off, the filtrate made up to 250 c.c., and the sulphur and chlorine determined in aliquot portions of this. The results obtained were: Insoluble matter, 20.2 per cent.; sulphur, 15.2 per cent.—corresponding to 33.6 per cent. calcium sulphide; chlorine, 29.3 per cent.—corresponding to 45.8 per cent. of calcium chloride: total, 99.6. This determination was made in a platinum crucible, but where the insoluble residue is not required porcelain is preferable.

Action of Peroxides on Organic Substances.—When barium peroxide is used as the oxidizing agent, the determination can only be made in a platinum crucible.

The decomposition of sodium benzene-sulphonate by means of barium peroxide and soda, and the subsequent addition of a little sodium peroxide, proceeds quietly, and the mass, when treated with dilute nitric acid, yields no sulphuretted hydrogen. Example: $C_6H_5 \cdot SO_3 \cdot Na$. 0.3498 gramme yielded 0.4625 barium sulphate = 18.10 per cent. sulphur. (Theory, 17.77 per cent.)

Sodium peroxide in the dry state acts so energetically on organic bodies that accurate results are impossible. On the other hand, its 3 or 4 per cent. aqueous solution has but little action either in the cold or on heating. This difficulty is overcome by the cautious evaporation of an aqueous solution of the sodium peroxide in contact with the organic substance, and under these circumstances the crucible is not attacked to any perceptible amount. The following method of procedure was adopted: The sodium benzene sulphonate was concentrated with as strong as possible a solution of sodium peroxide. When the mixture had attained the consistency of oil the crucible was heated over a very small flame until the mass began to darken. A few more drops of the peroxide solution were then added, and finally the crucible and its contents heated on the water-bath in a beaker containing a concentrated solution of sodium peroxide. After fifteen minutes the clear solution was acidified with hydrochloric acid, and the sulphur estimated by precipitation with barium chloride. Results obtained by this process:

		Sulphur.	
		Calculated, per cent.	Found, per cent.
Sodium benzene-sulphonate	...	17.17	17.88
Sulphanilic acid	...	18.50	18.21
Sodium aniline-sulphonate	...	13.86	14.10

Equally satisfactory results were obtained in the case of non-volatile chlorine compounds in alkaline solution, and of organic platinum chloride compounds. The weighed double platinum salt was introduced into a highly concentrated sodium peroxide solution, evaporated to dryness on the water-bath, more sodium peroxide

added, the crucible gently ignited, and finally heated on the water-bath in a beaker containing sodium peroxide solution. The separated platinum was filtered off, ignited and weighed, and the chlorine determined in the filtrate. Example: Iso-chinolin-benzyl-chloride, chloroplatinate.

	Calculated, per cent.	Found, per cent.
Platinum	23.0	23.1
Chlorine	25.0	24.9

The author considers that this method, which is applicable to all non-volatile chlorine and sulphur compounds, is a great improvement on the so-called Wallach method.

C. A. M.

Analysis of Copper and its Ores. Ledoux. (*Trans. Amer. Inst. Min. Eng.*, October, 1895; through *Chem. Zeit. Rep.*, 1895, 372.)—The author has been comparing the figures returned by a number of laboratories for the amounts of gold and silver existing in commercial coppers. He finds that the copper contained in the buttons of silver yielded by the dry process, which is usually assumed to compensate for the silver volatilized, varies very considerably, and bears no relation to the weight of silver lost. A combined process of wet analysis and assay is better, therefore, for the estimation of silver. This can be precipitated from solutions free from zinc and copper, and subsequently cupelled at a low temperature almost without loss. For gold this double process is not advisable, as some of the gold always dissolves during the solution of the copper in nitric acid, causing the results to be too low; dry assay is, however, satisfactory.

F. H. L.

Decomposition of Silicates by Boric Acid. P. Jannasch. (*Ber.*, 1895, xviii., 2822.)—One gramme of the finely-powdered silicate is mixed with 5 or 6 grammes of anhydrous boric acid, and ignited strongly for 15 or 20 minutes. The melt dissolves completely, or with a residue of silica, in hot water or alcohol. The solution is evaporated to dryness, strong hydrochloric acid and methyl alcohol added, and again dried down, when the boric acid volatilizes completely as methyl ester, the subsequent analysis being conducted in the usual manner.

F. H. L.

Three New Reagents for the Detection of Nitrites. M. G. Deniges. (*J. Pharm. Chim.*, 1895 [6], ii., 289; through *Chem. Zeit. Rep.*, 1895, 328.)—(1) By boiling phenol with Millon's reagent (mercuric nitrite and nitrate) a precipitate is formed, soluble with a red colour in acetic acid. This reaction will detect one part of phenol in two million, and, by a modification, the author has made it available as a test for nitrites. Two solutions are prepared: (a) 1 gramme of phenol, 4 c.c. of sulphuric acid, and 100 c.c. of water; (b) 3.5 gramme of mercuric oxide, 20 c.c. of glacial acetic acid dissolved in 100 c.c. of water, 0.5 c.c. of sulphuric acid added, and the whole filtered. Equal parts of the two liquids are mixed together, a few drops of the suspected liquid introduced, and boiled. A red colour will be formed in the presence of one part of nitrite per 2,000.

(2) Two c.c. of aniline are dissolved in 40 c.c. of acetic acid, and diluted to 100 c.c. with water. On boiling this solution with some of the liquid to be examined (from 1 drop to 10 c.c.), a coloration is produced varying from a straw-yellow to a deep orange, which, on cautious acidification, changes to a red. Chlorates and nitrates have no influence on the reaction, but hypochlorites, hypobromites, free chlorine, and bromine, as well as excess of acid, must be avoided.

(3) One gramme of resorcinol is dissolved in 10 drops of sulphuric acid and 100 c.c. of water. If 4 drops of the liquid under examination are shaken together with 2 c.c. of pure sulphuric acid and 5 drops of the resorcinol solution, the presence of nitrites is shown by a carmine or bluish-violet colour. This reaction will detect 0.01 milligramme of a nitrite.

F. H. L.

A Colour Reaction for Chlorates. M. G. Deniges. (*Journ. Pharm. Chim.*, 1895, [6], ii., 400; through *Chem. Zeit. Rep.*, 1895, 371.)—The resorcinol solution, mentioned in the preceding abstract, is also available for the detection of chlorates. A little pure sulphuric acid is mixed with the solution under examination, the whole cooled and shaken. Five drops of the resorcinol reagent are added, the mixture being only shaken again after immersion in cold water. Provided the solution does not contain more than 2 per cent. of a chlorate, a green colour is produced, recognisable when only 0.1 milligramme is present.

F. H. L.

Estimation of Iodine in the Ashes of Seaweed. H. Otto-Jensen. (Special Report to the Technical School at Bergen, 1894-95; through *Chem. Zeit. Rep.*, 1895, 371.)—The author has examined the three following methods for the determination of iodine in seaweed, and finds that Duflos's process—setting free the iodine with ferric sulphate, distilling it into iodide solution, etc.—is the quickest; but it is difficult to drive over the last traces of the halogen from the retort, the results, therefore, being usually too low. Lasseique's method—precipitation as palladium iodide—is very exact, but somewhat too delicate for technical laboratories. Gröger's process yields equally correct results, but the time taken in filtering and washing the manganese precipitate makes it slower than the first mentioned, although the use of the pump materially hastens the operation.

(Gröger's process consists in oxidizing the iodine by means of permanganate to the form of iodate, reducing the excess of the reagent by a few drops of alcohol, filtering off the manganese oxide, adding potassium iodide and hydrochloric acid to the filtrate, and estimating the iodine liberated with thiosulphate in the usual manner.)—ARS.

F. H. L.

Separation of Cobalt and Nickel. Carnot. (*Ann. des Mines*, 1895, vii., 624; and *Berg. u. Hüttenm. Zeit.*, 1895, liv., 370; through *Chem. Zeit. Rep.*, 1895, 371.)—Both metals are precipitated with bromine and caustic potash, the liquid boiled, the oxides washed, reduced to the metallic state in a stream of hydrogen, and, after another washing, are weighed together. They are dissolved in nitric acid, and the

solution divided into two portions. One is treated with hydrogen peroxide and potash, precipitating nickel monoxide and cobalt sesquioxide, the latter being then titrated with potassium iodide and thiosulphate. The other half is neutralized with potash, potassium cyanide added till the precipitate dissolves, then bromine; the cobalt remaining in solution as cyanide, while the nickel, coming down as sesquioxide, is titrated, in its turn, with iodide.

In mixtures containing little cobalt, the ammoniacal solutions may be treated with molybdate solution, the cobalt salt being practically insoluble in the cold liquid, while the double molybdate of nickel and ammonium is very soluble.

F. H. L.

Determination of Silicon in Iron. *Liebrich.* (*Stahl. u. Eisen*, 1895, xv., 1058; through *Chem. Zeit. Rep.*, 1895, 371.)—After the silica has been rendered insoluble in the ordinary manner, it is filtered off without much washing, introduced into a platinum crucible, the paper burnt, and three times its weight of acid potassium sulphate added. The crucible is laid on its side, heated gently till all the vapours are given off, then ignited more strongly, the blow-pipe being finally employed. All the graphite burns away, and, after cooling, the melt is taken up in very weak acid, when the silica will be found perfectly pure and white. The solution contains some iron and the titanium of the original sample.

F. H. L.

LEGAL.

IMPORTANT DECISION WITH REFERENCE TO MILK CERTIFICATES.

HIGH COURT OF JUSTICE.

(Before MR. JUSTICE HAWKINS and MR. JUSTICE KENNEDY.)

(Reprinted from the "*Times*" of January 28, 1896.)

FORTUNE VPELLANT—HANSON, RESPONDENT.

This was a special case stated by a stipendiary Metropolitan magistrate, and raised the question whether an analyst's certificate of milk ought to show what he considers to be the normal proportion of water in milk.

Mr. Macmorran appeared for the appellant, the inspector; Mr. Morton Smith for the respondent.

An information was taken out against the respondent at Clerkenwell Police Court. The offence charged was that he did sell and proceed to deliver on April 24, 1895, to Elias Jones a churn of milk in pursuance of a contract to sell, the same not being of the nature, substance, and quality of the milk demanded by the purchaser, in that it contained 5 per cent. of added water contrary to section 6 of the Sale of Food and Drugs Act, 1875, and of section 3 of the Act of 1879. The facts were these. The respondent sold a churn of milk to Jones on April 24. While it was being sold the appellant, inspector of nuisances for St. Mary, Islington, procured a sample of the milk and had it analysed by the public analyst. The certificate of the analyst was to the following effect:

"I . . . do hereby certify that I received on the 25th day of April, 1895, from Mr. Fortune a sample of milk marked as above for analysis (which then weighed 2½ oz.), and have analysed the same, and declare the results of my analysis to be as follows:

"I am of opinion that the said sample contained percentages of foreign ingredients as under: 5 per cent. of added water to the prejudice of the purchaser. Observations.—No change had taken place in the constitution of the article that would interfere with the analysis. This sample was handed to me undivided. I divided it into two parts, and one of which parts I returned to the purchaser."

This certificate was objected to as being bad evidence of the offence charged on the following grounds: (1) That it did not state as the result of the analysis the parts contained in the sample analysed; (2) that it contained statements of fact not authorized by the statutes, which facts should have been proved by sworn testimony and not by certificate. The statement of fact objected to was that he divided the sample into two parts, one of which he returned to the purchaser. In support of the first objection, it was contended that, as milk is composed partly of water, and there is no fixed standard, the analyst should state the exact quantities of the water and other constituent parts, so that a respondent might be able to determine whether he would require the inspector to attend for cross-examination, or whether he should appeal to the chemical officers at Somerset House. In support of the second point, it was contended that if the analyst were permitted to certify other facts than those authorized in the statute there would be no limit to the extent of proof which might be given by the certificate. The magistrate rejected the certificate, and therefore dismissed the information. He, however, granted this case at the request of the appellant. The form of the certificate is given in the schedule to the Act. It was followed for the most part in the certificate in the case—with the exception of the parts objected to. The following note appears in the schedule applicable to the word "observations": "Here the analyst may insert at his discretion his opinion as to whether the mixture (if any) was for the purpose of rendering the article portable or palatable or of preserving it, or of improving the appearance, or was unavoidable; and may state whether in excess of what is ordinary or otherwise, and whether the ingredient materials are or are not injurious to health. In the case of a certificate regarding milk, butter, or any article liable to decomposition, the analyst shall specially report whether any change had taken place in the constitution of the article that would interfere with the analysis."

Mr. Macmorran referred to the Food and Drugs Act, 1875, 38 and 39 Vict., c. 63, s. 18, and urged that the certificate was good. It was not necessary to set out the constituent parts of the sample. It was enough to show what foreign article was introduced. "*Bakewell v. Davies*" (1894) (1 Q.B., 296) showed that if there were words of surplusage they could be left out.

Dr. Morton Smith said that "*Bakewell v. Davies*" showed that if it was a case of abstraction the constituent parts need not be set out, but if it was a case of adulteration they must be. "*Newby v. Sims*" (1894) (1 Q.B., 478). It was most important to know what the analyst's basis of analysis was. It was all the more important in a case where only an addition of 5 per cent. of water was alleged. There was a dispute among scientific men as to what was the proper percentage of water in milk. Water was not a foreign ingredient.

The second point was not really contested.

Mr. Macmorran admitted that water was a constituent of milk, but pointed out that "added water" was a "foreign ingredient."

Mr. Justice Hawkins asked whether it was not the duty of the magistrate to decide the case. Ought he not, therefore, to be informed of the amount of water in milk which the analyst took as being the normal standard?

Mr. Macmorran said that the respondent could always cross-examine the analyst if he wished, and then the standard could be ascertained. The certificate was only *prima facie* evidence. In the case of "*Newby v. Sims*" the analyst had not followed the form of the

schedule. He cited "*Harrison v. Richards*" (45 J.P., 552) to prove that the justice ought to convict on the certificate of the analyst if it be not called in question.

The Court upheld the magistrate's decision.

Mr. Justice Hawkins went at length into the various sections and the form of certificate given in the schedule. He thought the certificate here did not conform to the requirements of the Act. It was admitted that milk naturally had water in it, and therefore it was necessary, in his opinion, that the magistrate should know what standard the analyst took. Standards differed greatly; the magistrate might take one, and the analyst another. To say there was 5 per cent. of "added water" was merely an opinion of the analyst. The justices ought to know upon what percentage he based his calculation. It was not as if water was a substance totally unknown in milk in its pure state.

Mr. Justice Kennedy concurred. The certificate was to give substantially the *data* on which the justices could act for themselves. It would be wrong to convict on a mere statement that there was 5 per cent. of added water. The analysis should be clear, and afford materials on which the justices and the accused also might know how the results had been arrived at. That was the principle of "*Newby v. Sims*" (1894) (Q.B., 478).

REVIEW.

MILK, ITS NATURE AND COMPOSITION. By C. M. AIKMAN, M.A., D.Sc. (London: Adam and Charles Black, 1895.) Price 3s. 6d.

The great number of papers referring to milk and milk-products which fill the pages of every volume of the ANALYST, and the lively discussions which have ensued whenever a paper of this kind was brought before the Society of Public Analysts, are sufficient proof of the great interest taken by analysts in the subject of milk and the preparations derived from it. A book treating on this subject is sure to attract the attention of analytical chemists among others, who, for one reason or another, wish to inform themselves about those widely-used substances milk, butter, cheese, etc. It was a happy notion of the author of the book before us "to give a short, popular statement of the more important facts of the chemistry and bacteriology of milk," as there did not exist a work in the English tongue dealing with the science of dairying. The author expresses the hope "that the work may possess interest for the general reader as well as for the farmer and the student of agriculture," and he might well have added "for the analyst," for the latter is very much helped in the interpretation and application of the results of his labour by some general knowledge of the nature, origin, and mode of manufacture of the materials he has to examine.

It is not an easy task to write a popular book which at the same time shall, to some extent, serve as a guide to the scientist. The author has well succeeded in solving the problem, although it might perhaps be said that the chemical part could have been somewhat shortened for the layman, while to the chemist it will not appear entirely satisfactory.

There is a good description given of the cow's udder, accompanied and made more intelligible by a number of well-drawn illustrations, and followed by a statement of our present views on the formation of milk. Speaking on the composition of milk, the Society of Public Analysts' standard is regarded as reasonable, although, as is

pointed out, genuine milk may occur which does not come up to that standard. The author holds "that a man selling such abnormal milk should be treated very much in the same way as a man selling adulterated milk." This opinion, which is shared by many public analysts, is highly objectionable. A man selling naturally poor milk may be made answerable for supplying an article not complying with the demands of the law, but he ought not to be punished as a criminal. The statement—in a footnote on p. 24—that in butter-fat glycerin is present to the amount of 4.5 per cent. is not likely to be accepted by chemists. There are a few other points to which objections might be raised, *e.g.*, the average amount of albuminoids present in milk being given as about 4 per cent.

The chapter "on the causes and conditions influencing the quality and quantity of milk" is very well written. On p. 59 the formation of a skin on milk heated to 50° C. is erroneously explained as caused by the coagulation of the albumin. The chief constituent of this skin is casein. The occurrence of slowly-rising cream is attributed to an extraordinary change of the nitrogenous matter, by which "a large proportion of the fatty globules becomes free." The meaning of this assertion is not clear.

Having regard to the very important part which micro-organisms play in dairying, as helpmates in the manufacture of butter and cheese, as adversaries in producing undesirable changes and decompositions, and as deadly foes in spreading diseases through the agency of milk, much space has been allotted to describe bacteria, their various kinds and forms, and their mode of development in general, and their action on milk, cream, butter, and cheese in particular. It is a most excellent chapter, which might be read with profit by everyone desirous of informing himself superficially on this topic. That the "salt taste" of milk is wrongly attributed to bacteria, and that in the list of cheeses which are inhabited by mould the famous Stilton has been omitted, cannot detract from the value of this chapter, which is well and profusely illustrated.

The following chapters treat on the scientific side of butter and cheese making; the facts are stated correctly, with few exceptions. As one instance of an error, it may be mentioned that the most favourable temperature for the action of rennet is given as 30° C., while it actually is 41° C. In enumerating methods for ascertaining the fat in milk, the latest processes have not been taken into account; and, speaking of the gravimetric determination, the well-known and widely, almost universally, adopted so-called coil process is attributed to Adam, instead of Mr. M. A. Adams.

The last chapter treats on "milk as food." Some people who are ordered to partake of a diet as easily digestible as milk, and who, however, have no particular liking for the latter, will be pleased to be informed that oysters are practically of the same nutritive value.

A number of German, Danish, French, and American works—in the first place Fleischmann's "Handbook on Dairying"—have been much drawn upon for the compilation of the book under review. The author freely acknowledges his indebtedness to the authors of those works, from which also a number of illustrations have been taken.

P. VIETH.

THE ANALYST.

MARCH, 1896.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday, February 5, 1896, at 8 p.m., in the Chemical Society's Rooms, Burlington House, the President (Dr. Stevenson) occupying the chair.

The minutes of the extraordinary meeting, held on January 8, and of the annual meeting, held on the same date, were read and confirmed.

The following gentlemen were elected: As member, Stevenson J. C. G. Macadam, F.I.C.; as associate, Charles Crocker.

The following gentlemen were proposed for election: As members, Richard A. Cripps, F.I.C., analyst, Hayward's Heath; Theophilus Vaughan Hughes, A.R.S.M., F.I.C., analytical and metallurgical chemist, assayer, and electrical engineer, Holywell, Flintshire; Thomas James Hutchinson, F.I.C., public analyst for Bury (Lancs.); and William Macnab, F.I.C., analytical and consulting chemist, 14, Great Smith Street, Westminster. As associate: E. T. Shelbourne, assistant to Dr. Alfred Hill, Birmingham.

Papers with the following titles were then read: "A Chemical Method of distinguishing between Cinnamon and Cassia." By C. H. Southwell. "The Determination of Oxygen in Commercial Copper." By Bertram Blount. "The Composition of Milk and Milk Products." By H. Droop Richmond. "A New Form of Carbonic Acid Apparatus." By Cecil H. Cribb, B.Sc. "Laboratory Notes." By Alfred H. Allen.

THE DETERMINATION OF OXYGEN IN COMMERCIAL COPPER.

BY BERTRAM BLOUNT, F.I.C.

(Read at the Meeting, February 5, 1896.)

THE only conventional method with which I am acquainted for the determination of oxygen in commercial copper consists in dissolving the metal in a solution of silver nitrate, and estimating the oxide of copper, which is supposed to be left as a basic nitrate, by solution in standard sulphuric acid and titration of the excess of acid by sodium carbonate. As commonly formulated, this method includes no precautions for ensuring regularity of action between the copper and silver nitrate, although, granting all other assumptions, such regularity is essential for the attainment of accuracy. In a recent investigation on the atomic weight of copper, the conditions requisite for the regularity of the reaction in question have been laid down. The

condition of most moment is that the solution shall be kept at a low temperature, preferably at 0°C ., for otherwise a smooth exchange between copper and silver does not take place, but an oxidation of the copper at the expense of the NO_3 group occurs, with the result that the residue of silver left after the reaction has been brought to a close contains basic cupric nitrate, not owing its presence to oxygen existing in the copper under analysis, but due to the nitrate of silver used as a reagent.

On observing the precaution of conducting the operation in a vessel buried in chips of ice, no difficulty arising from irregularity of interaction is experienced. Solution proceeds slowly, but eventually reaches the limit set by the relative quantities of the substances concerned. It is usual to employ a weight of copper somewhat greater than can be dissolved by the quantity of silver nitrate taken. A single strip of the metal is convenient, because the recovery and re-weighing of the undissolved portion is then an easy matter; when several small pieces are used, one may be overlooked in the mass of spongy silver which is precipitated. An additional precaution may be taken by filling the flask in which the reaction is carried out with CO_2 , so as to avoid possible oxidation of the copper during solution, and, for the same reason, using boiled-out water. The conscientious operator having done all this and more, soon becomes aware that the method is entirely worthless. In point of fact, the more carefully he excludes chance oxidation, the more erroneous are his results, for the method, when skilfully worked, fails to find any oxygen whatever in ordinary commercial copper, although that body is certainly present. The reason will be given in a later portion of this communication.

Having purged the silver nitrate method from accidental errors with the incidental discovery that its indications depended wholly on their occurrence, I turned my attention to other methods that seemed feasible on *a priori* grounds. Thus copper, in the form of thin turnings, was heated in a glass combustion-tube in a stream of hydrogen; the reduction was, however, only superficial. Next, copper, in the form of small bars, such as could be cut out of fire-box plate, was heated in an atmosphere of nitrogen, and sulphur vapour was then passed over it. Reaction readily occurred, and it was easy to convert the whole of the bar into a sulphide which approached the composition Cu_2S when the temperature was fairly high, but always contained rather more sulphur than corresponded with this formula. In adapting this method to practical requirements, the copper was placed in a boat in a combustion-tube, and the sulphur (recently fused) in another boat a little behind that containing the copper. Nitrogen, prepared from ammonium chloride and potassium nitrite, washed, dried, and purified by passage over red-hot copper, was caused to expel the air in the tube containing the boats in which were respectively the sulphur and the copper. When the displacement of air was complete, the portion of the tube in which was the boat containing the copper was heated, and then that in which was the sulphur boat. Sulphur vapour was driven gently by the stream of nitrogen over the heated copper, which forthwith began to glow. Excess of sulphur was caught in a cool part of the tube beyond the copper boat, and any flowers of sulphur that might be formed were filtered off through an asbestos plug. The exit end of the combustion-tube was connected with an absorption apparatus containing ammonia and hydrogen

peroxide. That portion of the sulphur which had been oxidized to SO_2 by the oxygen in the copper was caught in the absorption-tube, and finally estimated as barium sulphate.

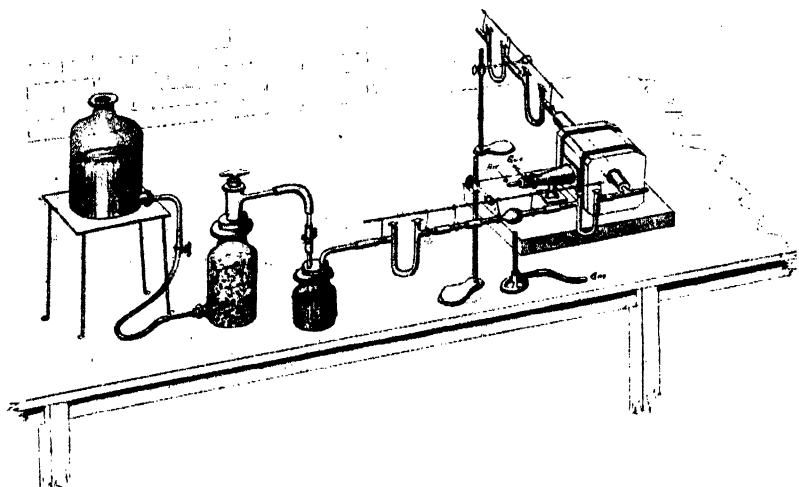
Owing to the ease with which the reaction between copper (even in pieces of large section) and sulphur takes place, a simplification of the above method was devised. A weighed rod of copper and an excess of sulphur (recently fused) were placed in a narrow piece of combustion-tube, closed at one end, which was then exhausted and sealed. On heating this tube to a temperature just short of the softening-point of the glass, the copper and sulphur combined; on the completion of the reaction, the tube was allowed to cool, and any gas which it contained collected over mercury and analysed.

Both methods yielded small and varying quantities of SO_2 , but as their working was improved and accidental presence of oxygen excluded, the quantity tended to a minimum. In short, much as in the silver nitrate method, the indications of the process disappeared when its errors were eliminated.

A little reflection allows these facts to be grouped under one head. The oxygen in commercial copper is generally assumed to exist as cuprous oxide dissolved in the metal. No doubt this is substantially true in ingot copper refined and containing no appreciable amount of impurity save oxygen, but not brought to tough-pitch. In copper intended for structural use, on the other hand, impurities other than oxygen, notably lead, are almost invariably present. There is no reason to suppose that the oxygen of such tough-pitch copper is present as cuprous oxide. Lead oxide or more complex bodies, such as lead arseniate, probably account for a considerable fraction. When copper containing oxygen in this form is dissolved in silver nitrate, the oxygen-bearing substances remain as a sludge with the spongy silver, and are substantially unattacked by the dilute standard sulphuric acid used to estimate the total basicity of the residue. The assumption on which the silver nitrate method is based is to the effect that all oxygen in the copper appears as basic cupric nitrate, a substance readily attacked by dilute sulphuric acid. Lead oxide or arseniate, on the other hand, in the refractory state which from their origin (in a mass of melted copper) they would naturally assume, would be but superficially attacked by weak sulphuric acid, and thus saturation of the acid in amount corresponding with the oxygen-contents of the copper could not occur. A similar influence appears to limit the reaction of copper containing oxygen in other forms than that of cuprous oxide when it is heated with sulphur. At the low temperature at which copper and cuprous oxide are attacked by sulphur, the more stable oxygen compounds—*e.g.*, lead oxide—are but little affected, and the oxygen is not eliminated as SO_2 . Doubtless it would be possible to decompose such oxides by means of sulphur at a higher temperature, but the practical difficulty caused by the softening of combustion-tubing at such a temperature then intrudes.

The method, after many trials, was abandoned, and a return made to the obvious and simple plan of reducing the copper in hydrogen. As mentioned above, only superficial reduction was obtained at any temperature below the softening-point of a glass combustion-tube. Fine division of the copper was thought undesirable, both from the risk of surface oxidation and from the difficulty of preparing filings or saw-

dust free from contamination. Recourse was had to the use of a temperature well above the melting-point of copper, the adoption of a porcelain tube set in a small injector furnace being therefore a necessity. Preliminary trials showed that it was not feasible to fuse copper in hydrogen and estimate the oxygen by loss of weight, for during the process the metal is spattered about with such violence that mechanical loss can hardly be avoided. It became necessary, therefore, to weigh the water produced by the action of hydrogen upon the melted copper, and the apparatus shown in the figure was finally adopted. The following are the principal parts: A hydrogen-generator containing zinc and sulphuric acid (not hydrochloric, lest vapours of that acid pass over with the hydrogen) is connected with a wash-bottle of copper sulphate solution followed by a sulphuric acid drying-tube. The oxygen of any air that may be entangled in the zinc or acid of the hydrogen-generator is eliminated by passing



the gas through a bulb-tube of platinized asbestos gently heated. Any water there formed is caught in a second sulphuric acid drying-tube, and the pure dry hydrogen (containing at worst a trace of nitrogen) enters a porcelain tube set horizontally in a small furnace of the injector pattern driven by a blast from a Root's blower (or bellows). The copper in which the oxygen is to be determined is cut in stout strips or bars to fit a small porcelain boat; 10 to 15 grammes may be conveniently taken. The porcelain tube, the boat, and all corks and connecting-tubes are thoroughly dried, and placed in position while so hot that they can scarcely be handled with comfort. Error due to adhering moisture is thus avoided. The exit end of the porcelain tube is connected with a weighed sulphuric acid tube protected by a sulphuric acid guard-tube. (I have laid emphasis on the use of sulphuric acid tubes throughout; they cannot be replaced by any less thorough means of drying; calcium chloride, as always for exact work, is "useless, dangerous, and ought to be abolished.") The inlet-tube is a \perp , with the right-hand horizontal limb closed by fusion. The vertical limb is that entering the rubber cork of the porcelain tube. This little

device makes it possible to look along the axis of the porcelain tube, and observe the behaviour of the copper during the process of reduction. The phenomena which occur are these: The copper suffers no visible change up to and including the time when it is at a bright red heat. No sensible action of the hydrogen upon the oxygen in the copper takes place up to this point, for no water appears in the exit tube. Just before fusion, when the metal is softening, small bubbles and excrescences appear on the surface of the copper, and evidently eject some gas. It seems probable that hydrogen is occluded by the copper at a temperature just short of fusion, and that the bubbles are due to the escape of water-vapour from the action of this hydrogen upon the oxygen of the copper. However this may be, it is certain that the appearance of these bubbles is immediately followed by the formation of water, which partly condenses in the glass exit tube, and is swept on into the weighed absorption tube in the usual manner. The bubbles soon cease to form, and the copper remains in tranquil fusion with a brilliant surface. The whole reaction is over in a couple of minutes, and the furnace can be shut down. Hydrogen is passed for another five minutes, to ensure the displacement of any water which may have lodged in the exit tube. During cooling, the copper again passes through a volcanic period, and finally solidifies with a rugose surface. This second spitting (which in outward form resembles the "sprouting" of silver) is probably due to expulsion of absorbed hydrogen; it is certainly not identical in nature with the first, for it is not attended by the formation of water. The violent motion of the melted copper during these two periods of disturbance causes a rain of tiny globules of copper, which coat the porcelain tube in the neighbourhood of the boat, and sometimes cause the two to adhere after cooling. This scattering of the copper is the effectual reason for the impracticableness of determining oxygen by loss of weight on heating in hydrogen.

Nothing now remains but to displace the hydrogen in the weighed tube with air, and to allow it to cool for weighing. Hydrogen is supplied continuously to the porcelain tube while it cools, so that all copper therein is brilliantly clean when the tube is opened. By thus avoiding casual oxidation the tube can be kept corked indefinitely, and used on the next occasion without any precaution save a preliminary warming.

I have had the method in use for a considerable time, and find it satisfactory in every way. It is applicable to copper-tin alloys, but not to brass, on account of the difficulty with which zinc oxide is reduced.

DISCUSSION.

Mr. BEVAN inquired what proportion of oxygen commercial copper usually contained.

Mr. HEHNER asked if Mr. Blount could suggest any explanation of the difficulty in obtaining a cuprous sulphide of constant composition to which he had referred. In Rose's well-known method for the determination of copper, which, as far as his experience went, always gave satisfactory results, pure Cu_2S was readily obtained.

Mr. BLOUNT said that the amount of oxygen present in commercial copper depended upon the proportion of other impurities. In a high-class tough-pitch copper containing over 99 per cent. of Cu, with, say, 0.15 per cent. of arsenic and a

similar quantity of lead, one might expect to find about $\frac{1}{10}$ per cent. of oxygen. In copper which was considerably under-poled, or in the condition in which it came direct from the refiners, the proportion of oxygen would be considerably greater.

With regard to Mr. Hehner's question, in the method which he (Mr. Blount) had described, the temperature was considerably lower than that reached in Rose's method; although a very fair approximation to Cu_2S could be obtained by carrying the heating as far as a combustion-tube would stand, the copper could not be said to be completely converted into Cu_2S ; an excess of sulphur always remained. For the purposes of the method described in the paper, it was, however, not necessary to take any special precautions to get the copper to that point.

A NEW FORM OF CARBONIC ACID APPARATUS.

By CECIL H. CRIBB, B.Sc. (LONDON).

(Read at the Meeting, February 5, 1896.)

THE apparatus is intended for the estimation of carbonic acid by the ordinary loss of weight method, and is identical in principle with the numerous forms at present in use. Of course anyone with a small flask, a cork or two, and some glass tubing can construct an appliance for himself which will answer every practical purpose, but that there is a demand for a more elaborate and specialized form of apparatus is obvious from an inspection of the catalogues of the makers of chemical apparatus, and from a glance round the shelves of most laboratories.

Quite apart from the merits or demerits of the method itself, which can only be regarded as one of moderate accuracy, most of the apparatus now in use suffer from more or less grave defects or inconveniences, and it is with a view of remedying some of these that the form represented in the diagram has been designed.

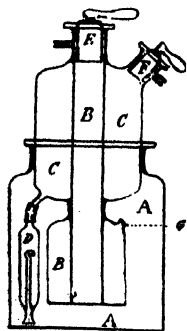
It consists of a wide-mouthed vessel, A, in which the substance to be analysed is placed, and an upper part composed of an absorption chamber, B, in which the carbonic anhydride is dried before escaping, together with a reservoir, C, for the acid used to decompose the carbonate.

To prevent any of the acid in C from prematurely reaching the carbonate, owing to the expansion of the air in C during the preliminary handling of the apparatus, a little trapped tube, D, is attached by indiarubber. It also serves another purpose, which is mentioned later.

The drying-chamber is charged by removing the stopcock, E, and dropping in the requisite quantity of sulphuric acid. Should too much be added, the excess may be poured out through G.

The central tube may with advantage be filled with broken up pumice-stone, or granulated calcium chloride may be used if preferred, as the desiccating agent.

To fill C with acid, the stopcock at F is taken out, and the little tube D is bent upwards parallel to the side of C, the "kink" in the indiarubber thus quite closing



the passage through D. The acid may then be poured in through F. When the stopcock is replaced and turned off, and D is allowed to fall down into its normal position, no liquid will leave the chamber, C, as long as the tap remains closed.

When thus prepared the apparatus is weighed, and the substance to be examined inserted through the wide mouth of A. It is then weighed again, and the decomposition of the carbonate is commenced by turning on first the tap at E and then that at F.

The following are the chief advantages of the apparatus :

The substance to be analysed is introduced with the greatest ease, and the residue left after the analysis can be completely recovered and used for other determinations.

The lower vessel can be cleaned out and wiped dry with a cloth, so that another analysis can be commenced in a few minutes.

The drying-chamber allows of the use of solid or liquid absorbents ; and in the case of the latter the length of the central tube renders any loss of acid, owing to a sudden evolution of gas, practically impossible if the tube be filled with broken pumice.

The comparatively large capacity of C, which holds over 25 c.c., enables a considerable weight of material to be operated upon. If desired, a measured quantity of standard acid could be employed, the residue in A being titrated back with standard alkali, thus giving a check on the gravimetric estimation without extra weighings.

If the little trapped tube is used, and is long enough to reach the bottom of A, the action becomes automatic, like that of a Kipp's apparatus, so that in commencing an analysis it is only necessary to turn on the taps at E and F, when the evolution of gas will proceed quietly until decomposition is complete, provided, of course, that the pressure of the column of liquid in C and D is more than sufficient to overcome the resistance interposed by the drying-chamber to the exit of the gas through E.

The two taps at E and F allow of a current of *dry* air being drawn through at the end of the analysis to remove residual carbonic anhydride, and also serve to shut off the drying-chamber completely from the external air when the apparatus is not in use.

The lower vessel, A, which is the part exposed to the greatest heat, is easily replaced in case of fracture.

The apparatus was made for me by Messrs. C. E. Müller and Co., of 148, High Holborn, from whom it can be obtained.

DISCUSSION.

Mr. HEHNER said that, while he acknowledged the ingenuity of the apparatus devised by Mr. Cribb, his experience led him to think that the old double-flask arrangement of Fresenius and Will was in no way surpassed by the many modifications of it, which seemed mainly designed to show the glass-blower's skill. He had come to the conclusion that, in order to obtain a reliable result with any arrangement of the kind, in which the carbon dioxide was measured by the loss in weight, so many precautions had to be taken that it was in every way preferable to determine

the carbon dioxide by absorption. Unless the evolution of the gas was very slow, and sulphuric acid tubes were joined to the apparatus both at the inlet and exit, correct results could only be obtained by chance. He did not think that it was worth while to bestow any thought upon the construction of new forms of the Schroetter device.

Mr. ALLEN said he thought the determination of carbon dioxide was liable to be very erroneous in any apparatus in which it was requisite to dry the gas by a considerable quantity of sulphuric acid. The amount of gas absorbed by the acid was much larger than was commonly supposed, and in some cases sufficient seriously to affect the accuracy of the results.

Mr. DIBDIN said that, from a somewhat extensive experience in the determination of carbonic acid in mortar, lime, etc., he had come to the conclusion that all methods of estimating this constituent by displacement were much to be mistrusted; and he preferred for exact determination to absorb it directly over mercury.

The PRESIDENT said he quite agreed that carbonic acid could be really satisfactorily determined only by absorption, although the ingenuity of Mr. Cribb's arrangement was certainly very praiseworthy.

Mr. CRIBB disclaimed any special admiration for apparatus of this kind, and said that as a matter of fact this modification had been designed in a fit of irritation at the shortcomings of the existing forms. The point mentioned by Mr. Allen as to the absorbing power of sulphuric acid, was entirely new to him. He thought, however, that it would hardly affect the results obtained by the method under discussion, inasmuch as the passage of a current of air through the acid would remove almost all the carbonic anhydride that had previously been absorbed. While not regarding the loss of weight method of estimation as one of great accuracy, he found it both convenient and rapid under certain circumstances, and thought that by the new form of apparatus both the speed and the accuracy of the process had been increased. Working without any special precautions, he generally found that duplicate estimations did not differ by much more than 0.1 per cent.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD ANALYSIS.

Estimation of Boric Acid in Milk. R. T. Thomson. (*Glasgow City Anal. Soc. Repts.*, 1895, p. 3.)—One to two grammes of sodium hydrate are added to 100 c.c. of milk, and the whole evaporated to dryness in a platinum dish. The residue is thoroughly charred, heated with 20 c.c. of water, and hydrochloric acid added drop by drop until all but the carbon is dissolved. The whole is transferred to a 100 c.c. flask, the bulk not being allowed to get above 50 to 60 c.c., and 0.5 gramme dry calcium chloride added. To this mixture a few drops of phenolphthalein solution are added, then a 10 per cent. solution of caustic soda, till a permanent slight pink colour is perceptible, and finally 25 c.c. of lime-water. In this way all

the phosphoric acid is precipitated as calcium phosphate. The mixture is made up to 100 c.c., thoroughly mixed and filtered through a dry filter. To 50 c.c. of the filtrate (equal to 50 grammes of the milk) normal sulphuric acid is added till the pink colour is gone, then methyl orange, and the addition of the acid continued until the yellow is just changed to pink. Fifth-normal caustic soda is now added till the liquid assumes the yellow tinge, excess of soda being avoided. At this stage all acids likely to be present exist as salts neutral to phenolphthalein, except boric acid, which, being neutral to methyl orange, exists in the free condition, and a little carbonic acid, which is expelled by boiling for a few minutes. The solution is cooled, a little phenolphthalein added, and as much glycerine as will give at least 30 per cent. of that substance in the titrated solution, and titrated with fifth-normal caustic soda till a distinct permanent pink colour is produced; each c.c. of fifth-normal soda is equal to 0.0124 gramme crystallized boric acid. A series of experiments with this process showed that no boric acid was precipitated along with the phosphate of calcium so long as the solution operated upon did not contain more than 0.2 per cent. of crystallized boric acid, but when stronger solutions were tested, irregular results were obtained. The charring of the milk is apt to drive off boric acid, but by carefully carrying the incineration only so far as is necessary to secure a residue which will yield a colourless solution, no appreciable loss occurs. W. J. S.

Relative Efficiency of Various Preservatives in Milk. R. T. Thomson. (*Glasgow City Anal. Soc. Repts.*, 1895, p. 5).—The author kept measured quantities of the same milk, to which the various preservatives were added, in stoppered bottles, under identical conditions, and examined them from time to time. The following table gives the result of the observations :

Preservative used.	Grains of Preservative per gal. of milk.	After standing 2 days.	After standing 4 days.	After standing 6 days.	After standing 7 days.	After 8 days. Lactic Acid, per cent.	After 11 days. Lactic Acid, per cent.
(Pure milk) ...	—	Distinctly turned	Slightly sour	Sour	Sour and curdled	·68	·71
Formic aldehyde (40 p.c.)	8.75 ·0125 p.c.	Sweet	Sweet	Sweet	Sweet	Sweet ·12	Sour & curdled ·43
„ „	17.5 ·025 p.c.	Sweet	Sweet	Sweet	Sweet	Sweet ·10	Sweet ·14
„ „	35 ·05 p.c.	Sweet	Sweet	Sweet	Sweet	Sweet ·07	Sweet ·10
Boric acid ...	35 ·05 p.c.	Sweet	Sweet	Turned	Sour and curdled	·42	·52
Boric acid + borax (calculated to boric acid)	35 (17.5 of ea.)	Sweet	Sweet	Sweet	Sweet	Sweet ·10	Sour ·32
Salicylic acid ...	17.5 ·025 p.c.	Sweet	Sweet	Sweet	Turned	Sour ·26	·42
„ „	35 ·05 p.c.	Sweet	Sweet	Sweet	Sweet	Sweet ·10	Sour ·33
Benzole acid ...	17.5 ·025 p.c.	Sweet	Sweet	Slightly turned	Sour	Sour ·45	·52

It will be seen that boric acid alone was not so effective as the mixture of boric acid and borax, the mixture being so made that each contributed 17.5 grains of

crystallized boric acid per gallon. Thirty-five grains of boric acid per gallon is the amount generally found in milks in which it is used. Formalin shows itself to be by far the most effective of the preservatives.

The author concludes with a few remarks on the physiological effects of the preservatives, in which he deprecates the practice of using them without having full proof that they are harmless, or without letting consumers know what they are getting. He quotes the following words of Dr. Leffmann: "Processes of digestion are allied to processes of decomposition, in so far that the latter are frequently preceded by transformation under the influence of ferments. We may infer, therefore, that whatever prevents putrefaction must at least delay digestion."

W. J. S.

Determination of the Dry Extract of Wine. L. Magnier de la Source. (*Ann. de Chim. Analyt.*, i., 7.)—The present method is proposed as a substitute for the author's process of evaporating the wine *in vacuo* without the application of heat where the operator does not possess the necessary apparatus for the production of a vacuum. For the modified process only a few instruments are required—a balance capable of weighing to a milligramme, a bell-glass fitting on a glass plate, a few cylindrical glasses 5 c.m. wide and 2 c.m. high, a pipette, and small quantities of sulphuric acid and phosphorus pentoxide, being sufficient to ensure results accurate enough for a.l. practical purposes.

Five c.c. of wine are taken. The glass containing the wine is placed under the bell-glass, and left there for about three days in presence of concentrated sulphuric acid. At the end of this time, the sulphuric acid dish is removed and substituted by one containing a little phosphorus pentoxide; another couple of days sufficing to complete the desiccation without the necessity of exhausting the receiver, thus saving the double operation generally practised.

To find the amount of the extract, the test-glass, previously tared, has then merely to be weighed, and the difference to be multiplied by 200 to express the result per litre. These agree fairly well with those from the vacuum process, and will be, with a little care, exact to within a margin of 0.5 gramme.

C. S.

TOXICOLOGICAL ANALYSIS.

The Toxicity of Acetylene. L. Brociner. (*Compt. Rend.*, 1895, cxxi., 773; through *Chem. Zeit. Rep.*, 1895, 416).—Blood dissolves about 0.8 of its volume of acetylene, most of which it loses on exposure to air in the cold, the rest at 60° C., while the whole disappears when placed in a vacuum. Examined with the spectroscope, the solution has no characteristic appearance, resembling rather normal oxygenated blood, and it is reduced by ammonium sulphide in a manner precisely similar to the latter. If there be any compound of acetylene and hæmoglobin, it must be very unstable. Acetylene cannot be considered any more poisonous than other hydrocarbons, such as methane, etc., for animals can survive its action even for many hours, if sufficient oxygen is present, and the atmosphere is renewed at intervals.

F. H. L.

Purification of Sulphuretted Hydrogen by means of Iodine. Z. H. Skraup. (*Zeits. österr. Apoth. Ver.*, 1896, xxxiv., 72; through *Chem. Zeit. Rep.*, 1896, 22.)—

Jacobson and Brunn's process for the removal of arsenic from this reagent, which consists in the employment of iodine, has been investigated by the present author, who finds that so long as not more than about 10 grammes of the gas are used in each test, the purification is sufficiently complete even for legal work. F. H. L.

ORGANIC ANALYSIS.

Saponification in the Cold. R. Henriques. (*Zeit. angew. Chem.*, 1895, pp. 721-724.)—The determination of the Köttstorfer and Reichert-Meissl numbers is in many ways simplified by carrying out the saponification in the cold, instead of by the usual process of boiling with alcoholic potash. From 3 to 4 grammes of the substance are dissolved in a flask with 25 c.c. of petroleum spirit, and treated with 25 c.c. of alcoholic soda of about normal strength. The saponification is complete in a few hours, but it is advisable to let the flask stand over-night, especially in the case of wool-fat and waxes. The titrating back of the excess of alkali is carried out in the usual manner. Where the soap has gelatinized, the flask should be gently warmed on the water-bath before titration, a little more alcohol being added if necessary.

Since waxes are only sparingly soluble in cold petroleum spirit, a slight modification of the process is necessary in their case. The substance is dissolved in warm petroleum spirit of a higher boiling-point (100° to 150° C.), the alcoholic potash then added, and the whole allowed to stand for 24 hours in the cold.

By using 3 to 4 grammes of the substance instead of the smaller amounts usually taken, the risk of errors introduced in measuring out and titration is considerably reduced. Another advantage of this over the old method is the avoidance of the alterations produced in strong alcoholic soda on continued boiling.

The following are saponification numbers obtained by this method and in the old way :

Oil, etc.	Cold Saponi- fication.	Warm Saponi- fication.	Oil, etc.	Cold Saponi- fication.	Warm Saponi- fication.
Linseed	195.3	195.0	Beef-margarine	201.5	205.7
"	195.0	193.5	Butter, I.	227.3	227
Rape, I.	178.6	178	Butter, II.	221.3	221
Rape, II.	176.4	175.6	Wool-fat	129.4	—
"	175.5	176.8	"	129.1	128.9
"	176.4	—	"	128.7	—
Castor, I.	186.0	185.9	Lanolin (anhy- drous)	90.4	90.7
"	186.3	—	Lanolin (anhy- drous)	90.6	—
Castor, II.	186.4	186.6	Yellow Bees- wax	96.6	—
"	185.8	185.8	Yellow Bees- wax	97.1	—
"	186.1	186.1	Yellow Bees- wax	97.4	—
Olive	196.3	196	Acetic Ether	640	—
"	195.4	196.6	"	636.6	Theory. 636.4
"	196.2	196.6	Malonic Ethyl Ether	700.5	—
"	196.6	—	"	700.7	700
Cotton	198.7	199.2			
"	199	200.0			
"	198.7	201.6			
"	198.7	—			
Cocoa-nut	266.8	266.4			
Beef-margarine	201.3	201			
"	201.2	202.2			

In adapting this method of saponification to the determination of the Reichert-Meissl number, the author gets rid of the objection that small quantities of the volatile acids remain in the form of their ethers and escape estimation.

Five grammes of the fat are dissolved in 25 c.c. of petroleum spirit in a covered porcelain dish, and allowed to stand over-night in contact with 25 c.c. of a 4 per cent. soda solution. Next morning the liquid is rapidly evaporated on the water-bath and the residue powdered. The author has never found that carbon dioxide was taken up during this process. The dry powder is then introduced into the distillation-flask, and the basin washed with the quantity of water requisite for the solution. The remainder of the process is as usual.

REICHERT-MEISSEL NUMBERS.

				Cold Saponification.		Old Method.	
				Found.	Mean.	Found.	Mean.
Berlin Butter, No. 1	30.6	30.6	30.0	30.1
"	"	30.4		30.2	
"	"	30.9			
Berlin Butter, No. 2	25.3	25.3	24.4	24.55
"	"	25.3		24.7	
"	"	25.3			
Königsberg Butter	26.2	26.0	25.1	25.4
"	"	26.0		24.4	
"	"	25.8		26.3	
Prime Margarine	1.20	1.27	25.8	0.93
"	"	1.34		0.91	
"	"			0.95	
Butter-mixture prepared in laboratory	8.9	9.1	8.8	8.65
Butter-mixture prepared in laboratory	9.3		8.5	

C. A. M.

A New Reagent for Bromine and Iodine. J. H. Kastle. (*Amer. Chem. Journ.*, xvii., 1895, pp. 704-708.)—The use of chlorine water as a reagent in testing for bromine and iodine is open to several objections which do not apply in the case of a new compound made by the author. This is the di-chlor derivative of benzene sulphonamide. It is prepared by dissolving benzene sulphonamide in as little as possible of a 1:10 solution of caustic soda and passing in a rapid current of chlorine. The white precipitate, which contains a considerable amount of unchanged amide, is filtered off, and the filtrate treated with the gas again. The second precipitate, which is very nearly pure, is filtered off and washed with hot water. It melts at 70° C, is almost insoluble in hot water, and in many of its physical properties resembles an acid chloride. It may be further purified by dissolving in alcohol and adding water which precipitates it in white lustrous plates. It acts on metallic bromides and iodides forming metallic chlorides, and the corresponding bromine and iodine derivatives of the amide. When this reaction is carried out in the presence of chloroform or carbon bisulphide, these solvents are coloured brown or violet, as in the ordinary test for bromide and iodine. In applying the test, the reagent may be

added either in the solid state or in carbon bisulphide solution. The author's experiments, which are given in detail, show that the test is capable of detecting 0·0000127 gramme of iodine in the presence of 0·0400 gramme of bromine, or 0·00000635 gramme of iodine in the presence of 0·003 gramme of bromine. When testing for very minute quantities of iodine, correspondingly small quantities of the reagent must be added, since it reacts with the liberated iodine to form colourless iodine trichloride.

C. A. M.

Spermaceti. Lyman F. Kebler. (*Amer. Journ. Pharm.*, lxxviii., 7.) The author has examined a number of samples of spermaceti from various sources, and found their melting-points to vary from 42° to 47° C., their specific gravities to range from 0·905 to 0·945 at 15° C., their saponification number to vary from 125·8 to 134·6, while the acid numbers ranged from 0 to 5·17, varying with the age of the sample. A fresh sample, recrystallized twice from alcohol (the cetine of Chevreul) melted at 44° C., had a specific gravity of 0·942, a saponification number of 127·6, and contained no free acid. The author considers that the acid and saponification numbers are the most reliable constants for spermaceti, and that adulterations would have to be most cleverly adjusted to disturb these without destroying the peculiar crystalline structure of the substance.

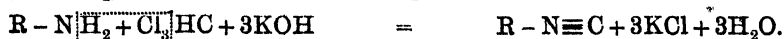
W. J. S.

Detection of Benzene in Petroleum Spirit, and vice-versa. Holde. (*Mittheil. K. Techn. Versuchss.*, Berlin, 1895, xiii., 241; through *Chem. Zeit. Rep.*, 1895, 416.)—The usual method for detecting the presence of either of these substances in the other, which consists in the observation of the colour produced by dissolving iodine in the liquid, is only available when large additions of the adulterant are suspected. The colours also vary according to the concentration of the solutions prepared.

A better process is based on the different solubility of asphaltum in the two liquids. The material is prepared for the test by washing its powder with petroleum spirit of the highest possible specific gravity, until it is so free from soluble matter that when a little fresh petroleum spirit is shaken with the powder in a test-tube it is only faintly coloured yellow. A little of the asphaltum is then placed in a filter-paper, a few c.c. of the liquid to be tested poured on it, and the colour of the filtrate noted. The presence of 5 to 10 per cent. of benzene in light petroleum of specific gravity 0·64 to 0·70, or of 10 per cent. of benzene in petroleum ether boiling below 35° C., can be detected, and, conversely, any considerable addition of petroleum to benzene is recognisable by the tint of the solution.

F. H. L.

Scheme for the Identification of Acetanilide, Phenacetin, etc. F. S. Hyde. (*Journ. Amer. Chem. Soc.*, xvii., 1895, pp. 933-935.)—One of the most important tests in this scheme is the "Carbylamine reaction," or "isonitrile test," made by boiling the substance with caustic potash and chloroform. The reaction occurs with primary amines ($R-NH_2$) in accordance with the equation:



SCHEME.

Solution of Substance in Water.

Pure Substance.	Melting-point C.	Solubility in Water.	Boiled with Excess KOH and a few drops of CHCl_3 .	Ferric Chloride.	Dilute Nitric Acid.	Bromine Water.
Acetanilide (phenyl acetamide): $\text{C}_6\text{H}_5\text{NH.COCH}_3$.	113°	Soluble in cold; more so in hot.	Odour of isonitrile.	Yellow solution; red on boiling.	Colourless.	White crystals; para-brom-acetanilide.
Exalgin (methyl phenyl acetamide): $\text{C}_6\text{H}_5\text{N.CH}_3\text{COCH}_3$.	101°	Not very soluble in cold; easily soluble in hot.	No odour of isonitrile; class of secondary amines.	Yellow solution; cloudy red on boiling.	Colourless.	No precipitate.
Phenacetin (acetyl-para-amidophenetol): $\text{C}_6\text{H}_4.\text{OC}_2\text{H}_5.\text{NH.COCH}_3$.	135°	Soluble with difficulty.	Odour of isonitrile.	Yellow solution; blood-red on boiling.	Cloudy-yellow solution. Crystals of nitro compound.	No precipitate.
Phenecoll hydrochloride (glycocoll-para-amido-phenetol): $\text{C}_6\text{H}_4\text{--}\begin{array}{c} \text{OC}_2\text{H}_5 \\ \text{NH.COCH}_2.\text{NH}_2.\text{HCl} \end{array}$.	No m.p. HCl comp.	Very soluble.	Odour of isonitrile.	Yellow solution; darkens and orange precipitate on boiling.	Colourless.	No precipitate.
Salol (phenol salicylate): $\text{C}_6\text{H}_4.\text{OH.COOC}_6\text{H}_5$.	43°	Soluble with difficulty.	No odour of isonitrile; yellow solution.	Yellow solution; blood-red on boiling.	Colourless.	No precipitate cold; white cloudy compound on boiling.
Resorcin (meta-dioxy-benzene): $\text{C}_6\text{H}_4(\text{OH})_2$.	118°	Easily soluble.	No odour of isonitrile; carmine-red solution.	Dark-violet; yellowish on adding drop of H_2SO_4 .	Yellow solution.	Yellowish precipitate dissolving immediately.
Quinine sulphate: $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_8.\text{H}_2\text{SO}_4$	Slightly soluble.	Base precipitates and dissolves on heating.	Yellow solution.	Colourless, with bluish fluorescence.	Dry substance on porcelain with weak bromine water gives green colour on adding two or three drops NH_4OH ; Thallioquin test.
Antipyrine (phenyl-dimethyl-pyrazolon): $\text{HOC}_3\text{N}_2(\text{CH}_3)_2\text{C}_6\text{H}_5$.	113°	Soluble.	Nothing.	Blood-red; disappears on adding drop of H_2SO_4 .	Colourless.	Yellowish white precipitate.

Phenacetin gives the isonitrile reaction, and therefore cannot be distinguished in this way from acetanilide. By boiling acetanilide with an alkaline solution of sodium

hypobromite (NaBrO), or with alkaline permanganate, the same smell of isonitrile is produced. In making the isonitrile test it is advisable to have the solution strongly alkaline, so as to ensure complete decomposition and a more perceptible odour.

C. A. M.

Determination of Nitrogen in the Compounds of Amines with Metallic Chlorides. W. von Dam. (*Rec. trav. chim. des Pays-Bas*, 1895, xiv., 217; through *Chem. Zeit. Rep.*, 1895, 415.)—As Delepine has already pointed out (*ANALYST*, xx., 111), Kjeldahl's process is useless in the case of platino-chlorides, owing to the production of free nitrogen. It works, however, perfectly with the compounds of gold and mercury chloride, although the reason of this peculiarity in the case of the gold salts is not apparent. By using a powerful reducing agent, such as 3 grammes of zinc-dust, well mixed with the amine platino-chloride before adding the sulphuric acid, the results are satisfactory; while the employment of mercury, except in the case of ammonium platino-chloride, also enables the process to yield correct results.

F. H. L.

The Examination of Soaps. Ed. Spaeth. (*Zeit. angew. Chem.*, 1896, 5-9.)—The usual method of estimating the "filling material" in a soap is to heat the dried soap with eight or ten times its volume of alcohol on the water-bath, filter through a tared filter, and weigh the residue. The principal objections to this are the solidification of the soap during filtration, and the difficulty of detaching all the insoluble matter from the beaker.

In place of this, the author proposes extraction in a Soxhlet extractor with alcohol, the soap being contained in a narrow glass vessel similar to a weighing bottle, but with perforations at the bottom and in the lid (see description, *Zeit. angew. Chem.*, 1893, 513). The holes at the bottom are covered with ignited asbestos fibre, over which is a layer of filter-paper, and finally a glass disc with fine perforations. The bottle thus prepared is dried at 105°C . for an hour, and weighed. From 5 to 6 grammes of the soap cut in thin strips are weighed into the bottle. This is then heated at from 50° to 60°C . to melt the soap, then for two hours on the water-bath, dried at 105°C ., and weighed. The loss in weight gives the amount of water in the soap.

The bottle without the lid is then placed in the Soxhlet extractor, and its contents extracted with neutral alcohol for about six hours. After complete extraction, the bottle and its contents are dried at 105°C ., and weighed, the result giving the amount of "filling material" in the soap. The determination of mineral substances, dextrin, gelatin, etc., in this is carried out in accordance with Benedikt's directions (*Anal. der Fette.*, 209).

Since the extraction is made with neutral alcohol, the amount of free fatty acids or alkalies in the alcoholic extract can be directly titrated with standard alkali or acid.

As a rapid process for judging of the value of a soap, the author has found the following reliable: About 5 grammes are weighed into a dried tared flask with a

mark at 120 c.c. The soap is dissolved in 50 per cent. alcohol, and, when cold, the solution is made up to the mark with the same alcohol. For the estimation of the water, 60 c.c. are pipetted into a platinum basin containing a known weight of ignited quartz sand, the alcohol gently evaporated, and the basin and its contents dried, first on the water-bath, with occasional stirring, and afterwards in the water-oven.

The remainder of the contents of the flask are again made up to 120 c.c. with the 50 per cent. alcohol, then poured into a separating funnel, the flask being washed out twice with 5 c.c. of the alcohol. The soap solution is shaken up with 20 c.c. of normal sulphuric acid, and again after the addition of 100 c.c. of a petroleum spirit of low boiling-point. After standing for some time, the lower layer is run off, the liquid filtered, and the excess of acid determined in an aliquot portion of the filtrate. From this can be calculated the amount of alkali corresponding to the fatty acids left in the separating funnel. The amount of glycerin may be estimated in another portion.

For the estimation of the fatty acids, 50 c.c. of the petroleum spirit solution left in the separating funnel are pipetted into a weighed flask, the petroleum spirit evaporated on the water-bath at 60° to 80° C., a current of hydrogen being meanwhile passed in. The flask is kept for ten minutes on the (boiling) water-bath, hydrogen being still introduced. It is then left for an hour over sulphuric acid, and weighed.

The saponification equivalent of the fatty acids in the flask is determined in the usual manner.

The comparative results of experiments tabulated by the author show clearly that the introduction of hydrogen during the drying of the fatty acids makes the figures obtained much more reliable.

C. A. M.

Quantitative Analysis of Soaps. W. Waltke and Co. (*Chem. Zeit.*, 1896, xx., 20 and 38.)—I. *Estimation of Inorganic Constituents.*—Benedikt has stated that soaps containing sodium silicate yield free alkali on treatment with alcohol, but the present authors deny the truth of this. Even when the samples contain as much as 15 per cent. of $\text{Na}_2\text{Si}_2\text{O}_7$, the alcoholic solutions are perfectly neutral.

From 5 to 10 grammes of the soap are extracted with boiling alcohol, the residue is dissolved in water, filtered, evaporated, and dried to constant weight. In the absence of borax, the silica is removed as usual, calculated into the above-mentioned silicate, and in the filtrate the total chlorine is determined volumetrically after removing the free acid. The silicate, reckoned as chloride, is subtracted from the total chloride obtained, the remainder being the amount of carbonate (expressed as chloride) existing in the soap.

In the presence of borax, the carbon dioxide is estimated in one portion of the aqueous solution of the salts. In the residue the silica and total sodium are found as before; when by deducting the carbonate and silicate (obtained from the yield of CO_2 and SiO_2 respectively), calculated as chloride, from the total sodium chloride, the remainder gives the amount of chloride corresponding to the borax in the original sample. The results quoted are satisfactory.

II. *Estimation of Uncombined Fat*.—Ten grammes of the soap, dried till free from water, are reduced to fine powder, and shaken for some minutes with about 100 c.c. of dry petroleum spirit. The mixture is diluted to 200 c.c., and allowed to stand till the supernatant liquid is perfectly clear. Fifty c.c. are then filtered through a double filter, run into a platinum basin, evaporated, dried at 110° C., and weighed. It very seldom happens that any soap dissolves in the ether if everything is quite dry, but the solubility of the extracted fat should always be tested with a fresh portion of the solvent, and if there is any insoluble matter, the analysis should be repeated.

F. H. L.

On Some Californian Oils. W. C. Blasdale. (*Jour. Amer. Chem. Soc.*, 1895, xvii., 935-941.)—This work was undertaken by the author mainly with the object of determining what were the limits of variation in the constants of Californian olive-oils. At the same time three new oils of vegetable origin were examined. For the investigation of the olive-oils eleven samples were prepared in the laboratory from six standard varieties of olives, and ten samples of commercial salad-oil were procured from different Californian makers. Of the latter three were found to be heavily adulterated with cotton-seed-oil, and one was very suspicious. The figures obtained with the pure olive-oils prepared in the laboratory were: Specific gravity, 0.9162-0.9174; refractive index, 1.4710-1.4717; thermal degree, 45.0-47.0; viscosity, 573-655; melting-point of fatty acids, 21°-26°; iodine absorption, 80.43-86.53; saponification number, 190.48-193.52; elaidin test, reaction in from $\frac{1}{2}$ to 6 hours; Milliau's test, no reaction.

Most of these constants agree fairly well with those obtained for European olive-oils, but the iodine absorption is higher than the commonly-accepted standard.

The thermal degree was obtained by noting the rise of temperature on mixing 15 c.c. of oil with 5 c.c. of concentrated sulphuric acid. The method of making the viscosity test was substantially that adopted by Babcock. Fifteen grammes of oil were saponified on the water-bath with exactly $7\frac{1}{2}$ grammes of caustic potash and 10 c.c. of alcohol. The soap was washed into an evaporating dish, heated to expel alcohol, and made up to 500 c.c. The viscosity of this solution was then determined by means of a torsion viscometer, and the result expressed in the number of grammes of sugar it would be necessary to add to a litre of water in order to produce a solution of equal viscosity. This method promises to be of great value in detecting adulteration in olive-oils. Although the variation among them is considerable, it is not so great as between olive-oil and many other oils. The author obtained the following figures with some of these: Cotton-seed, 280; almond, 645; rape-seed, 670; poppy-seed, 95; sesame, 415; lard-oil, 250; earth-nut, 220. Of these the only ones approaching olive-oil are almond, rape, and sesame oils. The first is unlikely to be used as an adulterant, and the other two are readily detected by other tests.

With regard to the elaidin test, the author is of the opinion that it is not as reliable as has been supposed.

The three new oils examined were:

1. Oil extracted with petroleum spirit from the fruit of the oak (*Quercus agrifolia*).

This was a deep brown fluorescent oil, solidifying at 10° C., and depositing waxy matter on long standing.

2. Oil from pine-nuts (probably *Pinus monophylla*). A^b brown, drying oil, of unpleasant smell and taste.

3. Oil from the fruit of the Californian nutmeg (*Tumion Californicum*).

The results obtained with these were :

	Specific Gravity.	Refractive Index.	Thermal Degree.	Viscosity.	Melting-point Fatty Acids.	Iodine Number.	Saponification Number.	Elaidin Test.	Millian's Test.
Acorn-oil ...	0.9162	1.4731	60.0	305	25°	100.66	199.26	no reaction	slight reaction
Pine-nut-oil ...	0.9333	1.4769	71.0	100	19°	101.3	192.81	brown colour no reaction	slight reaction
Californian nutmeg-oil ...	0.9072	1.4766	77.0	235	19°	94.71	191.30	no reaction	doubtful

C. A. M.

Determination of Hydrochloric Acid in the Gastric Juice. W. von Moracewski. (*D. med. Wochenschr.*, 1896, xxii., 24; through *Chem. Zeit. Rep.*, 1896, 23.)—The sample is evaporated on the water-bath down to 1 c.c., brought into a 100 c.c. flask, and made up to the mark with a mixture of 25 c.c. of absolute alcohol and 75 c.c. of dry ether. All the chlorides are precipitated, and the free hydrochloric acid in solution can, after neutralization, be determined by titration with silver, using potassium chromate as indicator.

F. H. L.

Analysis of Gutta-percha. J. A. Montpellier. (*Ann. de Chim. Analyt.*, i., 2.)—The durability of gutta-percha being in inverse ratio to the degree of oxidation or resinification, it becomes important to estimate the proportions of true gutta and the two resins (*alban* and *fluvial*) present; and as, furthermore, the water always present in the finished product facilitates oxidation, this also, as well as any impurities—whether introduced by accident or design—have to be determined.

To arrive at the amount of the pure gutta, 0.5 to 1 gramme of the substance is broken up into small pieces and placed in a tared filter, supported by a cone of platinum gauze. This is inserted in a flask containing absolute alcohol, so that the cone is immersed to half its height in the liquid. On surmounting the apparatus with a Soxhlet and a Liebig condenser, and heating on a sand-bath, the boiling alcohol extracts the resins, leaving the gutta behind. The last traces of the resins are removed by transferring the cone and its contents into the Soxhlet tube, where it is surrounded by alcohol vapour. Each section of the process requires five or six hours' boiling to accomplish, and the apparatus is allowed to cool after each stage.

The residue left on the filter must now be dried in a current of carbon dioxide at 100° C. To this end it is placed in a platinum or porcelain capsule, suspended in a tubulated balloon flask placed in an oil-bath. The carbon dioxide is generated in a

Kipp apparatus, and, after being purified by passage through a bottle containing potassium bicarbonate solution and one containing sulphuric acid, as well as through a glass containing pumice and sulphuric acid, is led into the flask, and emerges therefrom through the lateral tubule, carrying with it the water-vapour. This latter is absorbed by pumice and sulphuric acid in two U-tubes, followed by a 5-bulb Liebig tube containing sulphuric acid. At the end of five or six hours the rubber is re-weighed, the total loss, as compared with the original weight, indicating the percentage of resins and water. The water can be determined by the same apparatus, on a fresh portion (1 gramme) of the sample in which case the drying should proceed for six or seven hours.

The separation of the resins from each other is very difficult, and cannot be completely effected. An approximate determination can, however, be made by extracting 5 grammes of the gutta-percha in the manner already described, the alcoholic extract being then concentrated by evaporation at a gentle heat, until the limits of solubility of the *alban* are passed, whereupon this resin separates out on cooling, and leaves the other behind in the alcohol as a straw-coloured solution.

Impurities are estimated by dissolving out the gutta and resins by pure chloroform in the same apparatus as before, except that a constant level water-bath replaces the sand-bath. After washing, the residue, consisting of the impurities, is dried in a carbon dioxide oven.

The ash—which never exceeds 0.5 per cent.—is determined by weighing the residue left after incinerating a known quantity of the substance in a platinum or porcelain capsule.

C. S.

Estimation of the Total Tartaric Acid in Crude Tartar and Wine Lees.
D'H. de Rochefontaine. (*Ann. de Chim. Analyt.*, i., 25).—These substances are of somewhat complex constitution, consisting, in addition to the tartaric acid—present as acid tartrate of potassium and calcium tartrate—of calcium carbonate and sulphate, alumina, oxide of iron, silica, magnesia, chlorides, phosphates, organic and colouring matters, and particles of wood and sulphur.

For the estimation of their chief constituent two processes are in general use—the Marseilles method and the Goldenberg method. The former is of a less scientific character than the latter, but is sufficiently accurate for technical purposes.

Marseilles Method.—Place 50 grammes of powdered tartar or lees in a 300 c.c. porcelain capsule, and expose to the action of 100 c.c. of pure hydrochloric acid (20° Bé.) for three hours. Then add 15 grammes of pure powdered calcium carbonate, agitating at intervals until solution, which takes a considerable time, is effected. Add 100 c.c. of water, transfer to a 400 c.c. flask, fill up to the mark, and pass through a filter of not less than 25 cm. in diameter.

Transfer 191 c.c. of the filtered liquid into a conical glass, and neutralize with ammonia as exactly as possible (*i.e.*, until a violet tinge is imparted to red litmus-paper). The bulk of the calcium tartrate settles down immediately, but the whole must be left at rest for six hours, after which the clear liquid is decanted. This (the mother liquor) is allowed to stand for any further precipitate to come down while the principal precipitate is being treated.

Add 200 c.c. of water to the main precipitate, and leave to settle for a few minutes; then decant, reject the washings, and collect the precipitate on a tared filter 15 c.c. in diameter. Decant and reject the mother liquor, and join any precipitate which may have formed in it to the one on the filter, and wash twice with water. Then dry at 100° C. in an oven, and weigh, continuing the drying until two consecutive weighings at an interval of half an hour differ by not more than 0.15 gramme. The final weight (less that of the filter) multiplied by 2.3 gives the percentage of tartaric acid in the original substance.

The sources of error are the slight solubility of the calcium tartrate in water, especially in presence of a large excess of calcium chloride; the liability of iron and alumina to be precipitated by excess of ammonia; and the inclusion of a little colouring matter in the precipitate. These errors, however, compensate one another, and the method, if carried out strictly as prescribed, gives concordant results.

Goldenberg Method.—This well-known method is performed as follows: Place 3 grammes of the finely-powdered substance in a porcelain capsule, add 9 c.c. of pure hydrochloric acid of specific gravity 1.10, agitate, and leave to react for two hours; then add a small quantity of water, pass through a small filter into a 200 c.c. flask, wash till perfectly neutral, and make up to the mark. Transfer 100 c.c. of the solution to a porcelain capsule, and add 3 grammes of potassium carbonate little by little. Boil for half an hour, filter and wash, join the filtrate and washings, and evaporate down to 5 or 6 c.c. Then add 2 c.c. of 50 per cent. glacial acetic acid, allow to cool, and pour in 100 c.c. of 95 per cent. alcohol; agitate briskly, and allow the mixture to stand for half an hour. Collect the precipitate on a filter, and wash with alcohol until the washings are neutral. The filter with its contents are placed in the beaker used for effecting the precipitation, distilled water added, and the whole titrated with normal potassium hydrate solution until a drop changes the colour of red litmus-paper to blue. Each $\frac{1}{10}$ c.c. of alkali solution corresponds to 1 per cent. of tartaric acid in the original substance.

This method is more precise than the other, but the presence of colouring matter militates against accurate titration. It is advantageous to remove the filter before titrating. This is effected by driving off the alcohol at 40° to 50°, washing the filter with hot water (which dissolves the acid potassium tartrate), and titrating the liquid in the original precipitating glass.

C. S.

INORGANIC ANALYSIS.

Estimation of Sulphur and Carbon in Zinc. Robert Funk. (*Berl. Ber.*, xxviii., 3129.)—The author uses Fischer's reaction for the estimation of sulphur, which depends upon the formation of methylene-blue when hydrogen sulphide is allowed to act on a solution of *p*-amido-dimethylaniline and ferric chloride.

The zinc to be tested is placed in a flask holding about 200 c.c., having a tapped funnel ground into its neck. Pure hydrochloric acid is dropped upon the zinc, and the gas evolved passed through a Pettenkofer tube containing 25 c.c. of a mixture of equal volumes of a 2 per cent. solution of zinc sulphate and of a half per cent. solution of ammonia.

When the evolution of gas has ceased, the contents of the absorption tube are placed in a cylinder holding 40 c.c., the tube washed out with 2 c.c. of dilute hydrochloric acid, and the whole fluid made up to 40 c.c. One c.c. of the *p*-amidodimethylaniline solution (1 : 500) and one drop of a 10 per cent. ferric chloride solution are added. The blue colour produced is compared with that obtained under similar circumstances from a known amount of H_2S . The author finds that the purest zinc contains from 0 to 2.5 parts of sulphur in ten million parts of zinc.

The carbon is determined as follows: A strong combustion-tube is closed at one end, and bent to an angle of about 140° at a distance of about 8 cm. from its closed end. The shorter arm of the tube is filled with pure potassium chlorate, previously freed from organic matter by fusion; an asbestos plug is then inserted, followed by a layer of pure copper oxide. The zinc to be analysed is then inserted in a porcelain boat, and in front of this another layer of copper oxide. After the tube has been exhausted by a Sprengel pump, the heating is carefully commenced. The moment the pressure inside the combustion-tube equals that of the external atmosphere, a Pettenkofer absorption-tube, containing a 2 per cent. basic lead acetate solution (first recommended by Dupré and Hake) is attached, and the heating continued.

The author found that the sulphur and carbon are not dissolved in the zinc, but can be removed by melting the zinc and repeatedly filtering through an asbestos plug.

O. H.

Nessler's Reaction as a Test for Mercury or Iodides. G. Deniges. (*Chem. Zeit.*, 1896, xx., 70.)—The author remarks that the brown precipitate or coloration yielded by Nessler solution in presence of ammonia will serve equally well, *mutatis mutandis*, for the detection of mercury or for an iodide. For the former purpose, if the substance is already in solution, 2 c.c. of the fluid are taken; if a solid, a small amount is boiled with aqua regia, and evaporated down to a few drops, taken up with 5 c.c. of water, filtered if necessary, 2 c.c. of ammonia, and the minimum amount of potassium iodide required to dissolve the precipitate added. After shaking, a precipitate may form. Should this be coloured, it must be removed; but if it be white, or the liquid remain clear, the characteristic reaction will be obtained on the addition of caustic alkali. In the presence of copper, strong dilution is necessary. Excess of potassium iodide must be avoided.

When examining solutions for iodine which contain metals precipitated by the reagent, the test should be carried out on the filtrate from the ammonium sulphide precipitate of these. This is acidified, boiled till free from sulphuretted hydrogen, and made alkaline with ammonia. Caustic soda, and afterwards a few drops of mercuric chloride, are added, when the formation of a more or less dark red precipitate denotes the presence of iodine.

F. H. L.

The Volumetric Determination of Titanic Acid and Iron in Ores. H. L. Wells and W. L. Mitchell. (*Jour. Amer. Chem. Soc.*, 1895, xvii., pp. 878-883.)—A

volumetric method of determining titanous acid was proposed by Pisani (*Compt. Rend.*, lix., 289), but does not appear to have been found generally satisfactory. Marignac (*Zeit. anal. Chem.*, vii., 112) applied Pisani's method in the estimation of titanous acid in the presence of niobous acid, special conditions being adopted to avoid the reduction of the latter.

The authors have modified Pisani's process as improved by Marignac, and employ it for the determination of iron together with the titanous acid. Sulphuric acid solutions are used, and the liquid is protected from the air during cooling and titration by means of a current of carbon dioxide. The details of the operation are as follows: Five grammes of the pulverized ore are treated with 100 c.c. of concentrated hydrochloric acid in a covered beaker, using a gradually increasing heat, and adding more acid if necessary. When there is no further action, 50 c.c. of a mixture of equal volumes of sulphuric acid and water are added, and the liquid evaporated until it fumes strongly. After cooling, 200 c.c. of water are added, the whole heated until the sulphates dissolve, and the liquid filtered into a litre flask. If anything besides silicious matter is left on the filter-paper, it should be fused with potassium bisulphate, treated with concentrated sulphuric acid, and the sulphates dissolved in hot water and added to the main solution.

The liquid in the flask is made up to the mark with water, and 4 portions of 200 c.c. each taken, 2 in Erlenmeyer flasks (500 c.c.), and the other 2 in ordinary 350 c.c. flasks. Each of these represents 1 gramme of the ore.

To determine the iron, sulphuretted hydrogen is passed into the solutions in the ordinary flasks to saturation, after which they are boiled until all the sulphuretted hydrogen has been removed, care being taken to avoid any contact of the solution with the air by covering the mouths of the flasks with crucible lids. The flasks are then quickly filled to the neck with cold recently-boiled water, rapidly cooled, transferred to large beakers, and titrated with standard potassium permanganate.

To the solutions in the Erlenmeyer flasks 25 c.c. of concentrated sulphuric acid are added and 3 or 4 rods of pure zinc, about 50 mm. long and 6 or 7 mm. in diameter, are suspended in the liquid by means of a platinum wire attached to the loop of a porcelain crucible lid, which is inverted over the mouth of the flask. The liquid is then gently boiled for 30 or 40 minutes. Then, without interrupting the boiling, a rapid current of carbon dioxide is introduced under the cover. The flask is now rapidly cooled, the zinc washed with a jet of water and removed, and the solution titrated with permanganate, while the current of carbon dioxide is still being passed in. The difference between the permanganate used in this case and that required for the iron alone, represents the amount corresponding to the titanous acid. The factor for metallic iron divided by 0.7 gives the factor for titanous acid (TiO_2).

The most convenient strength for the permanganate solution is one of 7.9 grammes per litre, corresponding to about 0.014 gramme of metallic iron.

In the determination of iron by reduction with sulphuretted hydrogen, no effect is produced on cold permanganate solution by the precipitated sulphur present, but precipitated sulphides, such as copper sulphide, should be filtered off before boiling.

The results of test analyses of recrystallized potassium titanofluoride were as follows:

K_2TiF_6 taken.	Titanium found.	Titanium calculated.	Error.
Gramme.	Gramme.	Gramme.	Gramme.
0.7638	0.1437	0.1527	0.0090
0.7778	0.1524	0.1555	0.0031
0.6359	0.1215	0.1271	0.0056
0.4634	0.0882	0.0926	0.0044

The results were invariably too low. This was probably due partly to impurities in the salt used, but chiefly to the air gaining access to the flask in spite of all precautions. The accuracy of the method would apparently be increased by adding $\frac{1}{20}$ or $\frac{1}{30}$ to the amount of titanate acid found, but even without this correction it is likely to give more reliable results than those obtained by gravimetric determination.

C. A. M.

Quantitative Separation of Metals in Alkaline Solution by means of Hydrogen Peroxide. P. Jannasch, E. von Cloedt and H. Kammerer. (*Zeits. Anorgan. Chem.*, 1895, x., 405 and 408; through *Chem. Zeit. Rep.*, 1896, 5.)—To separate chromium from iron, manganese, or aluminium, Jannasch and von Cloedt add the solution containing the metals with 50 c.c. of 6 per cent. hydrogen peroxide and 30 c.c. of strong ammonia, in a pressure flask fitted with a wired-in rubber cork, and a tube drawn out to a capillary and sealed. After standing for an hour cold, the flask is heated on the water-bath for one or two hours longer. The chromium will all be found in solution, while the other metals are precipitated. The same process is convenient for the precipitation of manganese when mixed with zinc.

To separate arsenic from iron and manganese, Jannasch and Kammerer mix the liquid containing the latter metals together with arsenious acid with the alkaline peroxide, throwing down the resulting arseniate by magnesia mixture. Iron may also be separated from nickel, zinc and copper, by treating the chlorides, or, better still, the acetates, with the same reagent.

The peroxide should be prepared by Wolfenstein's process, *i.e.*, by distillation *in vacuo* and extraction with ether.

F. H. L.

Quantitative Separation of Chlorine and Bromine. S. Bugarski. (*Zeits. Anorgan. Chem.*, 1895, x., 387; through *Chem. Zeit. Rep.*, 1896, 5.)—The mixed chlorides and bromides are treated with 50 c.c. (or in presence of much of the latter 100 c.c.) of decinormal potassium di-iodate, 10 c.c. of 1 : 5 sulphuric acid, and enough water to bring the whole up to about 200 c.c. A little pumice is added, and the liquid boiled for 40 minutes, until the halogens are volatilized in the steam. The residue is made up to 100 c.c., some crystals of potassium iodide added, and the iodine determined in a portion of the liquid. From the amount of iodic acid used the bromine is calculated. The original liquid is titrated according to Volhard's method to give the total amount of the halogens, when, by difference, the chlorine can be found.

F. H. L.

Estimation of Phosphorus in Phosphor-Bronze. F. Oetell. (*Chem. Zeit.*, 1896, xx., 19.)—Fresenius' process (*Quant. Anal.*, ii., 550) for this determination is

very tedious, owing to the time taken up in filtering and washing the tin oxide containing the phosphorus, and if it is decomposed by fusion with sulphur and soda, instead of by boiling with acid, the product is contaminated with silica. Classen has suggested dissolving the oxide in sodium sulphide, converting the latter into the ammonium salt by means of ammonium sulphate, precipitating the tin electrolytically, and finally determining the phosphorus as usual. The large excess of ammonium salts, however, affects the accuracy of the method, and any arsenic in the bronze is estimated with the phosphorus.

From 3 to 10 grammes of the alloy are treated with nitric acid, the tin oxide filtered off and washed slightly. It is dried, ignited in a porcelain crucible, three times its weight of potassium cyanide added, and the mixture fused for some minutes. The tin is reduced, carrying with it any arsenic that may escape volatilization, while the phosphorus remains in the form of potassium salt. The mass is extracted with water, filtered, acidified with strong hydrochloric acid, boiled till free from hydrocyanic acid, and a current of sulphuretted hydrogen passed to remove traces of copper and tin. After filtering, it is boiled, a little bromine added, made alkaline with ammonia, and the phosphorus thrown down with magnesia mixture as usual.

The author remarks that the term "phosphor-bronze" applies rather to the process of manufacture than to the composition of the alloy, and that many samples contain extremely little phosphorus; while he considers that the amount present is of very little practical importance.

F. H. L.

New Reagents for Hydrogen Peroxide. L. I. von Nagy Ilosva. (*Ber.* 1895, xxviii., 2,029.)—Bach has described a new test for peroxide of hydrogen, consisting in a solution of aniline and potassium bichromate in presence of oxalic acid. The present author has found that a number of other aromatic amines, excepting diphenylamine and methyldiphenylamine, are also available, and that, of those examined, dimethylaniline is the most sensitive and convenient. Five drops of the amine and 0.03 gramme of potassium bichromate are dissolved in a litre of water, and, in testing, equal parts (5 c.c.) of the reagent and the liquid to be examined are mixed together, with the addition of a drop of 5 per cent. oxalic acid. A blank test is carried out at the same time with 5 c.c. each of water and the solution of the dimethylaniline, as the colour of the latter constantly changes, although this does not interfere with the delicacy of the reaction. Even in dilutions of one part per five million the presence of the peroxide is shown by a yellow colour appearing after a few minutes, while in stronger solutions a violet (methyl violet) colour is produced. Very weak solutions, containing an equivalent quantity of nitrous acid, are not amenable to this treatment, owing to the decomposition of the peroxide; and care must be taken that the coloration be not in any case caused by the action of ozone. The author notes that hydrogen peroxide does not entirely vanish from solutions of less than 1 per 500,000 in three or four days, and that stronger solutions (1 per 50,000) yield a faint reaction after a week.

F. H. L.

Estimation of Available Phosphoric Acid in Basic Slag. M. Gerlach and M. Passon. (*Chem. Zeit.*, 1896, xx., 87.)—Wagner's "dilute citrate solution" (*ANALYST*, xx., 215) contains per litre 14 grammes of free citric acid and 46 grammes combined as ammonium citrate. It is the former constituent which gives the liquid its power as a solvent, and Wagner's view that some of the phosphoric acid, when dissolved, acts as a solvent on the remainder cannot be substantiated. The presence of the ammonium citrate is to a large extent unnecessary, for of eighty-four slags treated with a 1·4 per cent. citric acid solution, all excepting three gave values agreeing with those obtained by Wagner's process. These three samples, which yielded 9·96, 13·02, and 8·82 per cent. of available phosphoric acid, gave up 12·10, 13·72, and 9·63 per cent. respectively to the free citric acid. On treating them, however, with a solution containing only one-tenth of the usual amount of citrate (18·6 grammes of crystallized citric acid and 1·17 grammes of ammonia per litre), perfectly concordant results were obtained; and the authors advise that the solution should always be made according to this formula. They also state that the speed of the agitating apparatus and the time of extraction have little influence on the results, but the temperature must be maintained at 17·5° C. It may be noted that by two or three successive extractions with fresh portions of the solvent the whole of the phosphoric acid present in the slag can be removed.

F. H. L.

An Improved Molybdate Solution. M. G. Meillere. (*J. Pharm. Chim.*, 1896 [6], iii. 61.)—By mixing together 200 c.c. of a 15 per cent. solution of ammonium molybdate, 20 c.c. of 1 in 1 sulphuric acid, and 30 c.c. of pure nitric acid, a liquid is obtained which is permanent for several months, and may even be heated to 100° C. without decomposition. When determining phosphoric acid, it suffices to start the precipitation by slight warming, allowing it to continue in the cold. In the case of arsenic acid prolonged warming is necessary, which may induce the deposition of molybdic acid; but if in the subsequent precipitation with magnesia mixture sufficient alkaline citrate (not tartrate) be present, this is without injurious effect.

F. H. L.

Estimation of Potassium. P. Lösche. (*Chem. Zeit.*, 1896, xx., 38.)—This process is specially applicable to such substances as carnallite, crude potassium salts, etc., where the amount of alkali present is approximately known. Fifty grammes of the sample are boiled with 150 c.c. of water and 10 c.c. of strong hydrochloric acid, and when cold diluted to 200 c.c. Ten c.c. of the filtrate are then treated in a small basin with about $1\frac{1}{2}$ times the theoretical amount of a solution of platinum chloride, containing 10 per cent. of the metal, necessary to effect complete precipitation. The whole is evaporated to dryness, broken up with a glass rod, and extracted with 96 per cent. alcohol. It is then placed on a filter, which has been dried at 120°—130° C., and weighed. Some 10 per cent. ammonium chloride is warmed to 30° C., and the precipitate washed therewith until all the sulphates are removed, the double chloride being perfectly insoluble. A final rinse in alcohol is given to remove the ammonium chloride, and the paper with its contents

dried again and weighed in the usual manner. A number of results obtained from different liquors containing potash, minerals, etc., are recorded, showing satisfactory agreement with the usual process of analysis, which involves the use of much larger quantities of platinum, beside the removal of the sulphuric acid.

F. H. L.

Standard Potassium Dichromate Solution. J. D. Dougall. (*Glasgow City Anal. Soc. Repts.*, 1895, p. 6.)—The author shows, by a series of experiments, that this solution may be made quite as, or even more, reliably by weighing the theoretical amount of potassium dichromate, and making up to a definite bulk, as by the more tedious process of first analysing a sample of iron wire and then standardizing the dichromate against this.

W. J. S.

Estimation of Lead Colorimetrically. Maurice Lucas. (*Bull. Soc. Chim.* (3) xv.-xvi, 39.)—For the determination of small quantities of lead, such as occur in copper, bronze, brass, tinfoil, water passed through lead pipes, etc., ordinary chemical methods are inefficient, and in such cases a colorimetric method may be resorted to with advantage. The sulphide is, however, the only lead salt suitable, the precipitates of all the others—and it is only the insoluble salts that exhibit colour—settling down too rapidly to admit of examination in this way. The presence of salts or alkalis affects the colour, alkalis imparting a brown, and neutral salts a grayish tinge, the latter being heightened by carbonates or bicarbonates; and as the collection of the precipitate is hastened in direct proportion to the quantity of salt or alkali and degree of concentration of the solution, their addition—with the exception of a slight quantity of alkali to prevent oxidation of the sulphide—is inadvisable. The sulphide, moreover, is six times, and twenty times, as sensitive as the chromate and iodide respectively, and permits the detection of as little as 0.00001 gramme—in fact, it is only surpassed in qualitative delicacy by microchemical methods. The strength of the solution most convenient for testing is between 1 and 20 mg. per litre.

In the analysis of bronze, which is given as a typical case, the nitric acid solution of 1 gramme of metal, freed from tin and antimony, is treated with 1 c.c. of sulphuric acid, evaporated down to a bulk of 7 or 8 c.c., taken up again with water and electrolyzed in a Riche apparatus with a constant current of 2 volts and 0.3 ampère for about twelve hours, after which the liquid is syphoned off and tested, to see that all the copper has been deposited; this also indicates the deposition of the lead.

The copper and lead adhering to the negative and positive poles respectively are washed while the current is still passing, and are weighed after drying quickly. The lead oxide multiplied by 0.865 will give the approximate amount of lead, and afford a guide to the dilution necessary: if over 5 mg. the lead should be weighed as sulphate without filtering.

Solutions of neutral lead nitrate (1 gramme), sodium hydrate (300 grammes), and sodium nitrate (640 grammes, per litre), having been prepared in advance, the lead oxide is redissolved in 1 c.c. of nitroso-nitric acid obtained by electrolyzing nitric acid. The crucible being washed with a little boiling water, and the solution neutralized by sodium hydrate from a Mohr burette, the volume is made up to 50 c.c. for each mg. of lead, and 5 drops of ammonium sulphide are mixed in.

The standard solutions for comparison must contain a quantity of sodium nitrate equal to the sodium hydrate added to the test solution, be of equal volume with the latter, and have 5 drops of ammonium sulphide added. When the solution is very dilute, it is better to divide it into two parts, and test one first. A number of standard solutions, graduated to twentieths above and below the shade observed, are then employed for comparison with the second half; this obviates the variation occurring in the shade on standing.

Between 0.0001 and 0.004 gramme the results are exact to within 10 per cent., and agree with sufficient precision. The method is more reliable than an estimation as sulphate, even if 10 grammes of substance are employed. C. S.

Analysis of Aluminium and its Alloys. H. Moissan. (*Comptes Rendus*, 1895, p. 851).—A preliminary examination for copper, by dissolving 2 grammes of the metal in dilute hydrochloric acid and passing a current of hydrogen sulphide, is necessary. If the amount of copper be very small, the liquid must be kept warm for several hours.

In the absence of copper, silicon is estimated by dissolving 3 grammes of the metal in dilute (1:10) HCl, any grayish residue left being fused with sodium carbonate in a platinum crucible, taken up with water and added to the main solution. After evaporation in a porcelain basin on the water-bath, the HCl is driven off by exposing the basin to a temperature of 125° for twelve hours. The sides of the basin are scraped at intervals with a platinum spatula, and the granular mass broken down by an agate pestle. The mass is then taken up with warm distilled water containing a little HCl, the solution boiled for a few moments, and the silica collected, ignited, and weighed.

For the estimation of aluminium and iron, the liquid from the preceding stage is made up to 500 c.c.; 25 c.c. (equal to about 0.15 gramme of metal) are then neutralized by ammonia in the cold, and precipitated by ammonium sulphide. After digesting for an hour the precipitate is collected, washed, dried, ignited, and weighed; this gives the total amount of oxide of iron and alumina. The iron is determined in 250 c.c. of the primary solution concentrated to 100 c.c., and treated with sufficient potassium hydrate (free from silica) to redissolve the alumina. After keeping the whole near boiling heat for ten minutes, the precipitate is washed five or six times, dissolved with diluted hydrochloric acid, re-precipitated by potash, washed and again dissolved, and the iron thrown down by ammonia. On filtering off and weighing the oxide of iron after ignition, the resulting weight deducted from that of the two oxides gives the amount of alumina.

As aluminium nitrate decomposes at a lower temperature than the corresponding sodium salt, the latter metal may be estimated by dissolving at a gentle heat 5 grammes of aluminium in nitric acid, diluted with an equal volume of water, the vessel being covered by an inverted funnel to prevent loss of the liquid. The solution is concentrated and evaporated to dryness, and the residue, after being powdered with an agate pestle, is maintained at a temperature below the fusing-point of sodium nitrate until the evolution of nitrous fumes ceases, whereupon a little boiling water is added and the alumina washed several times by decantation, the washings being then acidified with a few drops of nitric acid and evaporated to dryness. After taking up

several times with boiling water, to successively eliminate portions of the alumina mixed with the alkali nitrate, pure hydrochloric acid is added to the filtrate, and the liquid evaporated to dryness. The nitric acid is driven off at 300° , the sodium chloride re-dissolved, its chlorine determined as silver chloride, from which the weight of sodium can be deduced.

Carbon is estimated by triturating 2 grammes of metal with 10 to 15 grammes of powdered mercuric chloride and a little water. The mixture, after evaporation, is placed in a porcelain boat, inserted in a Bohemian glass tube, which is heated to redness, and through which a current of oxygen (free from CO_2) is passed. The effluent gas is absorbed by KHO, and the carbon calculated from the increase in weight of the potash tubes.

In alloys of copper and aluminium, the weight of the former metal is ascertained by dissolving 0.5 gramme in nitric acid free from chlorine, diluting the solution to 50 c.c., and passing a current of 0.1 ampère for six hours at 60° , or twenty-four hours in the cold, the copper being washed, dried, and weighed as metal.

The remaining components are, after throwing down the copper by hydrogen sulphide, estimated as already described.

The following is an analysis of Pittsburg aluminium :

Al	98.82
Fe	0.27
Si	0.15
Cu	0.35
Na	0.10
C	0.41
N	traces.
Ti	traces.
S	nil.

100.10

C. S.

APPARATUS.

Liebig Condenser. G. W. A. Kahlbaum. (*Berl. Ber.*, xxix., 69.)—The condenser bearing Liebig's name was invented by Christian Ehrenfried Weigel, and described by him in his dissertation of March 25, 1771. (Liebig was born in 1803.) Weigel's condenser was described in Goettling's "Manual of Chemistry," 1794, and Liebig, when he first described the use of this condenser, mentioned the source from which he had obtained the description of the apparatus.

O. H.

An Improved Gas-measuring Vessel. F. Cochiu. (*Chem. Zeit.*, 1896, xx., 89.)—This is an ordinary gas burette, which may have its upper portion expanded or not. The top of the tube terminates in a neck, into which is ground a perforated stopper, which may either carry a funnel, a small connecting-tube, or a three-way tap. To the under side of this stopper a thermometer is attached in such a position that the latter is entirely within the measuring-tube. With this device the temperature of the gas can be read off at once, without having to wait for some time until it has fallen to the temperature of the outside air. The contrivance is especially useful for the collection of warm gases.

F. H. L.

THE ANALYST.

APRIL, 1896.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday evening, March 4, the President (Dr. Stevenson) occupying the chair. The minutes of the previous meeting were read and confirmed.

The following gentlemen were elected: As members—Richard A. Cripps, F.I.C., Hayward's Heath; T. Vaughan Hughes, F.I.C., Holywell, Flintshire; T. J. Hutchinson, F.I.C., Public Analyst for Bury (Lancs.); and William Macnab, F.I.C., Westminster. As associate—E. T. Shelbourn, assistant to Dr. Alfred Hill, F.R.S.E.

The following gentlemen were proposed for election as members: F. W. Richardson, 2, Farcliffe Place, Bradford, Consulting Chemist to the Bradford Corporation; and Walter Thorp, B.Sc., Limerick, Consulting Chemist to the Irish Dairy Association.

The following papers were read: "On the Estimation of the Diastatic Power of Malt," by Walter J. Sykes, M.D., and C. A. Mitchell, B.A.; "Further Note on the Detection of Formalin," by H. Droop Richinond and L. K. Boseley; "The Detection of Formalin," by Otto Hehner; "Note on the Detection of Cotton-seed Oil in Lard," by E. J. Bevan.

The following pieces of apparatus were exhibited by Mr. E. J. Bevan: A Zeiss refractometer, and a calculating machine.

NOTE ON THE TITRATION OF QUININE.

By ALFRED H. ALLEN.

(Read at the Meeting, February 5, 1896.)

In titrating alkaloids much depends on the indicator employed and the method of applying it. Methyl-orange, rosolic acid, iodeosin, phloxin, phenol-phthalein, gallein, lacmoid, brazil-wood, logwood, litmus, and cochineal have all been employed and have found advocates.

The behaviour of the alkaloids and organic bases with indicators of neutrality has been very imperfectly studied. It is frequently stated that a certain alkaloid is distinctly alkaline (presumably to litmus), but it is only rarely and of recent years that chemists appear to have attempted to estimate alkaloids by titration with standard acid. Where this is desired, phenol-phthalein is quite inapplicable. Litmus answers in some cases, but by no means invariably. With methyl-orange, in the majority of cases a determination of tolerable accuracy and a fairly sharp end-

reaction are obtainable. Cochineal, brazil-wood, and logwood are often useful indicators.

In titrating quinine, an anomaly occurs which has misled more than one observer. This arises from the fact that the ordinary quinine sulphate of commerce, having, when anhydrous, the formula $(C_{20}H_{24}N_2O_2)_2 \cdot H_2SO_4$, though practically neutral to brazil-wood, logwood, and cochineal, is strongly alkaline to methyl-orange. The point of neutrality when titrating quinine with cochineal, brazil-wood, or logwood, is therefore reached when sufficient acid has been added to convert the quinine into the sparingly soluble sulphate of the formula $Qu_2 \cdot H_2SO_4$; whereas in the case of methyl-orange the end-reaction corresponds to the formation of the readily soluble acid sulphate of the formula $Qu \cdot H_2SO_4$. Hence twice the volume of standard acid will be required by a given weight of quinine when methyl-orange is employed, as when brazil-wood, logwood, or cochineal is used as the indicator. If nitric acid or hydrochloric acid be substituted for sulphuric acid, the results are similar, the salts $Qu \cdot 2HNO_3$ and $Qu \cdot 2HCl$ being neutral to methyl-orange. The sparingly-soluble sulphate, $Qu_2 \cdot H_2SO_4$, is distinctly alkaline to litmus, and hence this indicator cannot be conveniently used for the titration of quinine, though the end-reaction is well marked.

The fact that $Qu_2 \cdot H_2SO_4$ is neutral to brazil-wood, and $Qu \cdot H_2SO_4$ to methyl-orange, is parallel to many other instances, the most interesting being that of the phosphates of sodium. Thus:

NaH_2PO_4 is neutral to methyl-orange.

Na_2HPO_4 is neutral to phenol-phthalein.

Na_3PO_4 is neutral to "Poirrier's blue, C.L.B."

The foregoing statements are a record of facts observed independently in my laboratory, and carefully verified by numerous experiments. The main fact, however, was previously recorded by Seaton and Richmond (ANALYST, xv. 43), who state that they "found that quinine bisulphate is neutral to methyl-orange, while the base itself has no action on phenol-phthalein," and they based on these facts a process for the determination of quinine in medicines. Their instructions assume that the medicine originally contained excess of acid, and was free from any inorganic base precipitable by baryta. Seaton and Richmond give figures showing that the method is fairly accurate when applied to pure sulphate of quinine, but the conditions under which the titrations are performed in practice render the method of little value. It is useless in presence of citric and other organic acids, and the colouring matter present in quinine wine prevents the end of the reaction from being accurately observed.

Owing to no formula for "quinine bisulphate" being given by Seaton and Richmond, nor the consequences pointed out, the fact on which their process was based was misinterpreted by me ("Commercial Organic Analysis," vol. iii., part ii., p. 403, footnote), and apparently has been quite overlooked by other writers.

Experiments made in my laboratory on the titration of cinchonine and cinchonidine with various indicators had led to such anomalous results as to render it doubtful if the constitution of these bases, or at least the composition of the commercial article, is correctly understood.

My thanks are due to Mr. A. B. Searle for the execution of the experiments referred to in this note.

DISCUSSION.

The PRESIDENT said that Mr. Allen had stated very clearly and concisely the facts as to the titration of the quinine alkaloids. The parallel drawn between quinine compounds and phosphates was especially interesting. It was easy to understand how readily errors might be made in quinine estimations, in consequence of the nomenclature of the quinine salts. A similar instance of ambiguity had occurred in the case of the two chlorides of mercury.

NOTE ON THE PREPARATION OF PURE HYDROFLUORIC ACID.

By ALFRED H. ALLEN.

(Read at the Meeting, February 5, 1896.)

COMMERCIAL hydrofluoric acid is liable to contain various impurities, including lead, iron, aluminium, calcium sulphate, organic matter, etc. In the absence of a platinum still it is not easy to prepare a strong acid suitable for use in the analysis of silicates, but the necessity of obtaining a product free from fixed impurities led me, some years since, to adopt the following contrivance, which I have since used with very satisfactory results.

The apparatus simply consists of the largest platinum crucible available. In this is placed a mixture of ordinary hydrofluoric acid with about an equal measure of strong sulphuric acid. In a smaller platinum crucible is placed a weighed quantity of the silicate to be treated, and this is moistened with three or four drops of strong sulphuric acid. The smaller crucible is then placed inside the larger, and is preferably supported on a disc of plaster of Paris, or on a ring cut from a piece of one-inch leaden gas-pipe. The larger crucible is then covered with a platinum dish filled with cold water. On heating the arrangement on an iron plate, the hydrofluoric acid in the outer crucible is volatilized, and is condensed on the lower surface of the platinum dish, from which it drops on to the silicate contained in the inner crucible. After a time, when about 10 c.c. of distillate has collected in the inner crucible, the latter is removed, the contents evaporated off, and the treatment once or twice repeated, to ensure the complete decomposition of the silicate.

By the foregoing means pure concentrated hydrofluoric acid is prepared at the time and in the exact quantity required, without the use of any special apparatus.

DISCUSSION.

Mr. BLOUNT said that he had distilled absolutely pure hydrofluoric acid in a leaden vessel, having an exit tube sloped upwards, and connected with a platinum condensing-tube sloped downwards, so that the condensed acid came into contact with platinum alone. This was a very convenient plan, as a fairly large quantity of the acid could be made at one operation.

Mr. ALLEN said that when a small quantity of lead in the hydrofluoric acid was of no consequence, he had himself used a leaden retort constructed out of gas-pipe, but without the platinum tube mentioned by Mr. Blount.

THE COMPOSITION OF MILK AND MILK PRODUCTS.

By H. DROOP RICHMOND.

(Read at the Meeting, February 5, 1896.)

THIS paper gives a *résumé* of the work done in the laboratory of the Aylesbury Dairy Company during the year 1895.

Of the 30,068 samples analysed during 1895, 24,662 were milk samples, and the mean composition of 11,081 received from the farms is given in Table I.:

	A.M. Milk.				P.M. Milk.				Mean.			
January ...	1·0325	12·76	3·87	8·89	1·0323	13·15	4·23	8·92	1·0324	12·95	4·05	8·90
February ...	1·0326	12·72	3·81	8·91	1·0324	13·16	4·23	8·93	1·0325	12·94	4·02	8·92
March ...	1·0326	12·48	3·60	8·88	1·0324	12·88	4·00	8·88	1·0325	12·68	3·80	8·88
April ...	1·0324	12·49	3·65	8·84	1·0322	12·86	4·03	8·83	1·0323	12·68	3·84	8·84
May ...	1·0327	12·40	3·50	8·90	1·0323	12·74	3·90	8·84	1·0325	12·57	3·70	8·87
June ...	1·0323	12·21	3·43	8·78	1·0319	12·49	3·78	8·71	1·0321	12·36	3·60	8·76
July ...	1·0320	12·14	3·45	8·69	1·0315	12·51	3·87	8·64	1·0317	12·33	3·66	8·67
August ...	1·0318	12·16	3·49	8·67	1·0314	12·60	3·96	8·64	1·0316	12·38	3·72	8·66
September ...	1·0321	12·24	3·51	8·73	1·0316	12·59	3·91	8·68	1·0319	12·41	3·71	8·70
October ...	1·0326	12·67	3·77	8·90	1·0321	13·15	4·30	8·85	1·0323	12·91	4·04	8·87
November ...	1·0326	12·74	3·83	8·91	1·0322	13·10	4·24	8·86	1·0324	12·92	4·03	8·89
December ...	1·0327	12·63	3·71	8·92	1·0324	12·97	4·08	8·89	1·0326	12·80	3·89	8·91
Average ...	1·0321	12·47	3·64	8·83	1·0321	12·84	4·03	8·81	1·0322	12·66	3·84	8·82

The results are given to show the difference between the morning and evening milks.

No cases of the fat falling below 3·0 per cent. in the mixed milk from any farm have been observed during the year.

The mean composition of the milk of the goat and the ass is:

	Goat.	Ass.
Total solids ...	13·24	10·23
Fat ...	3·78	1·18
Sugar ...	4·49	6·86
Proteids ...	4·10	1·74
Ash ...	·87	·45

As the composition of cream produced by separators is wholly dependent on the adjustment of the separator, the average composition of cream has no scientific import, and the table which has been given in former years is omitted.

The analysis of one sample of unusually thick cream, which was made for experimental purposes, was as follows:

Total solids ...	68·18
Fat ...	64·88
Ash ...	·28
Solids-not-fat ...	3·30

Ratio of 100 parts of water to solids-not-fat = 10·4.

This cream was analysed to see whether the view put forward in a former paper (ANALYST, xix., 73), that the ratio of water to solids-not-fat was the same in cream as in milk, and therefore that the fat did not carry any proteids, was borne out in cream of great thickness. Were it a fact that the fat did carry proteids, the ratio of

solids-not-fat to water should be distinctly higher in very rich cream than in milk. It was, however, found that the ratio was identical with that in the milk used for its preparation.

The composition of clotted cream was as follows :

		Maximum.	Minimum.	Average.
Total solids	...	73.77	59.01	66.81
Fat	...	66.89	48.27	58.21
Ash	...	1.17	.49	.71
Solids-not-fat	...	11.70	5.85	8.60

The ratio of the ash to solids-not-fat = 1 : 12 is the same in clotted cream as in milk.

The bulk of the fat estimations in all products was made by the Leffmann-Beam method ; the Gerber method, in which the chemical principles of the Leffmann-Beam method have been incorporated, has also been used, on account of the manner in which the machine employed lends itself to modification. Two centrifugal machines, one having a steam turbine fitted, and the other a water motor, have been employed with very satisfactory results as compared with hand-driving gear.

The following Reichert figures have been found in butter during the year :

	Maximum.	Minimum.	Average.		R.W. Figure.
French butter	14.35	12.4	13.28	× 2.2	= 29.2
Irish butter	13.7	11.5	13.00	„	28.6
Australian butter	15.2	13.4	14.17	„	31.2

The percentage of water has been as follows :

	Maximum.	Minimum.	Average.	
French fresh butter	18.00	12.25	14.78	
„ salt	14.43	10.25	12.97	
Australian salt butter	15.72	9.09	12.82	
Irish butter	15.54	11.84	13.68	
English fresh butter, I.	16.26	11.87	13.79	direct from churning.
„ „ „ II.	15.71	12.50	13.54	24—48 hours old.
„ salt „ I.	18.06	11.77	14.74	direct from churning.
„ „ „ II.	16.63	10.30	13.33	24—48 hours old.
„ „ „ III.	15.09	9.69	12.00	10—30 days old.

The percentage of water was also calculated for the summer and winter months respectively :

	Summer.	Winter.
French fresh butter	14.46 per cent.	15.09 per cent.
English butter	14.17 „	13.33 „ (all kinds).

The following tables show the frequency with which amounts of water differing by 1 per cent. were found :

FRENCH FRESH BUTTER.

Per Cent. Water.	No. of Samples.	Per Cent. on Total.
18—19	1	.2
17—18	6	1.3
16—17	32	7.1
15—16	137	30.4
14—15	190	42.1
13—14	83	18.4
12—13	2	.4
Total	451	99.9

FRENCH SALT BUTTER.

Per Cent. Water.	No. of Samples.	Per Cent. on Total.
14—15	7	14·6
13—14	23	47·9
12—13	9	18·8
11—12	7	14·6
10—11	2	4·2
Total	48	100·1

The English fresh butters (all churned at Bayswater) were divided into two classes :

I. Samples taken immediately after churning.

II. „ „ after a lapse of 24 to 48 hours.

		I.		II.	
Per Cent. of Water.	No. of Samples.	Per Cent. on Total.	No. of Samples.	Per Cent. on Total.	
16—17	1	1·7			
15—16	6	10·3	2	10	
14—15	18	31·3	3	15	
13—14	19	32·8	7	35	
12—13	12	20·7	8	40	
11—12	2	3·4			
Total	58		20		

The English salt butters were similarly divided into three classes :

I. Samples taken immediately after churning.

II. „ „ after a lapse of 24—48 hours.

III. „ „ „ „ 10—30 days.

		I.		II.		III.	
Per Cent. of Water.	No. of Samples.	Per Cent. on Total.	No. of Samples.	Per Cent. on Total.	No. of Samples.	Per Cent. on Total.	
18—19	1	1·3					
17—18	2	2·5					
16—17	12	15·0	3	3·8			
15—14	18	22·5	4	5·1	1	3·6	
14—13	20	25·0	10	12·6			
13—12	20	25·0	27	34·1	3	10·7	
12—11	6	7·5	30	38·0	8	28·6	
11—10	1	1·3	4	5·1	12	42·9	
10—11	—	—	1	1·3	3	10·7	
9—10	—	—	—	—	1	3·6	
Totals	80		79		28		

It was found by experiment that the loss of water from salt butter was practically complete after the first week, and it is quite legitimate to average butters from 10 to 30 days old.

The following conclusions are drawn :

I. French butters, both fresh and salt, have contained fully 1 per cent. more water than in former years (see former reports).

II. From the experiments on English butters it is seen that fresh butter loses

a very small amount of water on keeping; salt butter, on the contrary, loses water very rapidly, especially during the first day or two after making, and more slowly during the next week, when the loss nearly ceases. It appears that the loss of water from fresh butter is due chiefly to evaporation, and the loss of water from salt butter chiefly to brine running out. It is interesting to note in connection with this that duplicate analyses of fresh butter (taken from different parts of a lump) have never differed more than 0.26 per cent., while differences of 0.7 per cent. have been observed with salt butter.

III. French fresh butter contains more water in winter than in summer; English butters contain more water in summer than in winter. The number of English butters prepared in winter was, however, small (66), and of these only four were prepared during the winter proper (December to February); the average of these four was 14.27 per cent., or .1 per cent. higher than the summer average.

It seems that exceptionally high temperatures and exceptionally low temperatures have a tendency to cause butter to contain a high percentage of water.

IV. Taking butter from 24 to 48 hours old to represent commercial butter, it is seen that fresh butter on the average contains more water than salt butter.

It appears from the tables above that when sampled directly after churning, salt butter contains more water than fresh; but this conclusion is not wholly justified. It is known that certain conditions of the cream churned are favourable to a low percentage of water in butter, and the cream used for churning fresh butter fulfilled those conditions better than that used for churning salt butter. In the few cases where both fresh and salt butter were made from the same cream, the amounts of water were nearly identical.

There appears to be little foundation for the commonly accepted statement that salt butter contains more water than fresh butter.

V. It has been observed that, broadly speaking, the nearer the percentage of water is to 13.5, the better is the quality of the butter, and that when the limits of 12 per cent. on one hand, and 15 per cent. on the other are overstepped, there is, in a large number of cases, a marked falling off in quality. This rule is, however, far from absolute.

It is known that water is added to butter as an adulterant, and there is no method of distinguishing between water fraudulently added to butter and that naturally found therein. One chemical method and two so-called "practical" methods have been put forward. The chemical method is based on the fact that butter contains a certain amount of solids-not-fat, which would be reduced by adding water; Vieth (*ANALYST*, xvi. 1) was, I believe, the first to calculate the ratio of the solids-not-fat to 100 parts of water, and to point out that in unwashed butters there were 10 parts of solids-not-fat to 100 of water, while washed butters contained 5 parts; he also pointed out that the variations were considerable.

But for the fact that this method has been seriously recommended to public analysts by a butter dealer, Mr. Gibson (*Report Food Products Adulteration, Blue Book*, No. 363, 2078-2079), it would be hardly necessary to give quantitative results.

I have taken a series of 109 samples of butter known to be without addition of

water (including some insufficiently worked), and have calculated the proportion of solids-not-fat to 100 parts of water; the results are contained in the following table :

S.-n.-F. to 100 parts Water.	No. of Samples.	Percentage of Water.		Average.
		Max.	Min.	
14	1			10.28
11-12	1			13.48
10-11	1			12.87
8-9	6	13.64	11.30	12.61
7-8	16	14.97	10.19	12.82
6-7	26	14.64	11.08	12.87
5-6	31	15.33	10.36	12.94
4-5	19	19.23	11.01	14.06
3-4	7)	14.74	11.90	13.41
2-9	1)			

I took 90 parts of a sample of butter which contained 12.5 per cent. water, and mixed 10 parts of water with it; it gave the following figures on analysis :

Water	21.06 per cent.
Fat	75.84 " "
Salt	2.46 " "
Solids-not-fat64 " "
Ratio of solids-not-fat to 100 of water	3.0 " "

Though I believe any public analyst would have condemned this sample, solely on account of the amount of water present, the ratio of solids-not-fat to 100 parts of water does not fall below the minimum found in genuine butters, and it would have to be passed by the proposed test.

I need not further comment on the method.

FURTHER NOTES ON THE DETECTION OF FORMALIN.

By H. DROOP RICHMOND AND L. K. BOSELEY.

(*Read at the Meeting, March 4, 1896.*)

In a former paper on this subject (*ANALYST*, xx. 154) an error has crept into the text with regard to the number of molecules of ammonia which react with formaldehyde. Legler states that 4 molecules of ammonia react with 6 of formaldehyde, and Lösekan maintains that 6 molecules are equal to 6 of formaldehyde.

In the same number of the *ANALYST* (p. 157) Rideal states that the formalin used for milk preservation contains 2 ounces of formaldehyde to the gallon, or 1 : 320. This ratio is an error, the true ratio being 1 : 80 (or 2 : 160). He states also that $\frac{1}{2}$ pint of this solution is added to the churn of seventeen to eighteen gallons, *i.e.*, 1 part diluted formalin to 288 parts milk. From these data he concludes that 1 part of formaldehyde is contained in 46080 parts of milk, and a retro-calculation shows that he has neither used the correct ratio of 2 ounces to the gallon = 1 : 80, nor his assumed ratio 1 : 320, but a ratio of 1 : 160.

Rideal also states that all milks give a pink colour when mixed with Schiff's reagent, and concludes that milk contains a non-volatile aldehydic compound. He

has, however, completely overlooked the fact that aldehydes do not give a pink colour with Schiff's reagent, but a reddish violet one; while alkalies, either free or combined with a weak acid, give a pink colour. We find that in the presence of an acid, even when very dilute, the pink colour is not developed in milk, while the inhibitory effect of dilute acid on the colour given by aldehydes is negligible. Indeed, if Schiff's reagent is prepared according to Caro's directions with rosaniline, sodium sulphite, and hydrochloric acid, it always contains an excess of hydrochloric acid.

On p. 167 of the same journal an abstract of a paper by Weigle and Merkel is given. As a proof that formalin has an adverse influence on peptic digestion, they adduce the fact that an addition of formalin to milk renders the casein insoluble in pepsin and hydrochloric acid. As without the addition of formalin the insoluble casein-dys-peptone is formed, it is difficult to see that their observation proves that formalin renders the albuminoids of milk less digestible.

We have studied the comparative delicacy of the reactions employed for detecting formalin. Of the general aldehyde reactions, we find that the reduction of silver nitrate is, though very delicate, of so general a character as to be misleading, even when following Tollen's directions to test in the cold with a mixture of 3 grammes silver nitrate dissolved in 30 c.c. of ammonia (specific gravity .923), and 3 grammes sodium hydroxide in 30 of water (*Ber.*, xv. 1635).

Schiff's test is also very delicate, but must be performed in a slightly acid solution. We do not find that milk-sugar, even after *boiling* with dilute sulphuric acid, gives any colour, and consider that it is quite safe to test the whey produced by adding dilute sulphuric acid to milk, especially as the test, not being characteristic, is only of a preliminary kind. The troublesome distillation is avoided in this way.

We have not thought it necessary to investigate the delicacy of the para-diazo-benzene-sulphonic acid test, the resorcinol test, nor the nitro-prusside test.

We find that the delicacy of Hehner's reaction may be much increased by diluting the milk with an equal bulk of water and adding sulphuric acid of 90 to 94 per cent. strength. Under these conditions milk, in the absence of formaldehyde, gives a slight greenish tinge at the junction of the two liquids, while a violet ring is formed when formaldehyde is present. This colour is permanent for two or three days. In the absence of formaldehyde a brownish-red colour is developed after some hours, not at the junction of the two liquids, but lower down in the acid. It cannot be mistaken by anyone who has had any experience with the test for the formaldehyde reaction. By using this modified test, the use of milk is more convenient than that of peptone when testing in aqueous solution (*e.g.*, a distillate). The delicacy of Hehner's test is about equal to that of Schiff's.

Trillat's reaction with dimethylaniline is less delicate, but does not require any distillation. The lead oxide mentioned in our former note should be lead peroxide (PbO_2). The blue colour is not very stable.

Plöchl's test fails with very small amounts of formaldehyde.

We find that our test with diphenylamine is not of very great delicacy. Though we had no difficulty in getting a reaction with milk to which the amount of formalin solution prescribed by the vendors was added, we find that Hehner's and Schiff's tests give distinct reactions when ours fails. The statement that the precipitate is

coloured green if the acid used contained nitrates is incorrect. We find that when acid free from nitrates is used, the green coloration still appears on continued boiling.

Of about equal delicacy is Trillat's aniline test. It is necessary to operate in the cold, as the precipitate is easily soluble in hot water, being again deposited on cooling. The precipitate given by acetaldehyde with aniline is very much more soluble than that given by formaldehyde.

By boiling a solution of hydrazine sulphate, to which a slight excess of caustic soda solution has been added, with formaldehyde, formalazine is produced as a yellowish-white flocculent precipitate. This reaction is of less delicacy than any of the foregoing.

We have attempted the preparation of thio-meta-formaldehyde as a test for formalin, but without success in dilute solutions, though Wohl states that it is insoluble.

We prefer to rely on Hehner's test (with the modification mentioned above) for the detection of formalin in milk, and to use Schiff's test (in the whey produced by sulphuric acid) and Trillat's dimethylaniline test to confirm, and if the quantity appears large from the above reactions, to distil the milk into diphenylamine dissolved in a slight excess of sulphuric acid, and boil.

THE DETECTION OF FORMALIN.

BY OTTO HEHNER.

(Read at the Meeting, March 4, 1896.)

SCHIFF'S reagent as a test for formaldehyde is far from satisfactory. Apart from the fact that it reacts with all aldehydes, and is, therefore, not applicable to fermented beverages or fluids, such as wine or vinegar, which contain naturally traces of acetaldehyde, or to food-substances which by fermentation might yield traces of acetaldehyde, it acts but very slowly when very small quantities of formaldehyde have to be looked for, such as would be present in milk when preserved with formalin in accordance with the instructions furnished by the vendors. If these instructions are complied with, about one part of formaldehyde only is present in about sixty thousand parts of milk. Schiff's reagent is quite capable of indicating such traces, but only after some hours, or when the distillate from the milk plus Schiff's reagent has stood over night. As pointed out by Richmond and Boseley in their recent paper (*ANALYST*, xx. 154), when an excess of sulphurous acid is used in the preparation of the reagent, no reaction is obtained with traces of formaldehyde, while "the red coloration appears on warming Schiff's reagent, on blowing air through it, or even on placing it in an uncorked bottle." I can fully confirm these statements, and can further add that Schiff's reagent may give a red coloration on standing for a short time even with pure distilled water in stoppered cylinders, or with a distillate from milk which is perfectly free from formalin. Thus, if to 25 c.c. of water only one or two drops of Schiff's reagent are added, a red colour appears already in half an hour,

the amount of oxygen contained in the water being sufficient to oxidize the sulphurous acid contained in the added reagent, even though the mixture has been placed in a stoppered cylinder filled to the neck. When a little more Schiff's reagent is added—say ten drops—the coloration does not appear. It follows that an analyst who is not aware of this circumstance—and so far attention has not been drawn to it—may in one of two samples of milk, both equally free from formalin, apparently find formalin in the one and not in the other, although he may have apparently operated under precisely the same conditions in both.

I find that the best way of operating is to add about five drops of the reagent to the distillate from 100 c.c. of milk (amounting to about 25 c.c.), place the mixture in a stoppered cylinder, to observe the colour next morning, and then to add a few drops of sulphurous acid solution. After a short time any colour which may be due to oxidation will have vanished, while that due to the presence of an aldehyde remains. There is certainly a difference in the tint produced by colour oxidation, which resembles that of rosaniline, and that of the aldehyde compound, which is violet; and with those small traces with which we have often to deal, only a comparison of the relative colours would allow of anything like a safe conclusion being drawn.

From the experience I have collected I am clearly of opinion that Schiff's test is a dangerous one, and should only be used as a confirmation.

I have made some experiments as to the rate at which formalin disappears from milk on keeping. To three portions of pure milk formaldehyde was added in the proportions of 1 of the aldehyde to 25,000, 1 to 50,000, and 1 to 100,000, the milk thus preserved being kept in stoppered bottles. After one week no formalin could be detected in the sample containing 1 part in 100,000; after two weeks none in the 1 in 50,000 sample; while after three weeks there was only the faintest trace discoverable in the 1 in 25,000 sample. These experiments were made in January, when the temperature was low, the Schiff's reagent being used with the precautions described.

The reaction which Messrs. Richmond and Boseley have described in the paper alluded to (*loc. cit.*) as having been first pointed out to them by myself, and which depends upon the blue colour produced when milk containing formaldehyde is mixed with sulphuric acid, is infinitely more characteristic than Schiff's reaction, especially when the test is applied in the form given it by Mr. Richmond, where the milk to be tested is allowed to float upon the surface of sulphuric acid, anhydrous, or nearly so, contained in a test-tube. I find that in this way one part of formaldehyde can be readily detected in 200,000 parts of milk. Acetaldehyde does not give this reaction, and as no distillation is required, the test is of the simplest possible nature as regards milk. I cannot bear out the statement of Messrs. Richmond and Boseley that it is possible to obtain a blue coloration with formaldehyde to which some peptone solution had been added. The peptones which I used, and which undoubtedly were rich in peptone, gave no blue coloration. Egg-albumen does give the reaction in the presence of formalin, but so feebly as to lead me to conclude that it is not the albumen itself, but some trace of impurity contained in it, which enters into the reaction. Casein precipitated from milk, re-dissolved in alkali, and again precipitated and dissolved, gives the colour most strongly. Milk-whey also gives it, but gelatin, as already pointed out by Richmond and Boseley, does not. I have tried

many other substances likely to be present in milk, but have found nothing so satisfactory as casein.

To test wine or vinegar for formaldehyde, I add a drop of milk to the sample, and pour the mixture carefully on the surface of strong sulphuric acid contained in a test-tube. A blue ring appears at the zone of contact when formalin is present, while ordinary aldehyde gives no reaction.

The test is also well fitted for the detection of formaldehyde in butter, the aqueous liquor resulting when the butter is melted for analysis being used.

The following is another equally sensitive and characteristic test. If to the distillate from a sample of milk, etc., one drop of a dilute aqueous solution of phenol is added, and the mixture poured upon strong sulphuric acid contained in a test-tube, a bright crimson colour appears in the zone of contact. This colour is still readily seen with one part of formaldehyde in 200,000 of water. If there is more than one part in 100,000, there is seen above the red ring a white, milky zone, while in stronger solutions a copious white or slightly pink, curdy precipitate is obtained. This reaction has the advantage of the one above referred to, inasmuch as it is obtained with formaldehyde solutions of all strengths, while the blue colour with milk is not obtained with milk containing much formaldehyde. I have not ascertained the upper limit of sensitiveness of the reaction, but the blue colour is *only* obtained with traces.

Acetaldehyde also gives a coloration and a precipitate with phenol and sulphuric acid, but it is orange-yellow, not crimson.

Many other hydroxy-derivates of benzol, such as salicylic acid, resorcinol, and pyrogallol, give the red colour with formaldehyde. Hydroquinone does not give the red colour, only an orange-yellow one.

The reaction only succeeds when carried out as described above; the phenol must first be mixed with the solution to be tested and the mixture poured upon the sulphuric acid. Only a trace of phenol must be used. If the hydroxy-compound is first dissolved in the acid, and the formaldehyde solution then added, no colour is obtained.

Of course it is very well known that formaldehyde most readily causes the condensation of aromatic substances. Thus, the tannins readily condense in the presence of hydrochloric acid, forming insoluble compounds. Richmond refers to Kleeberg ("Annalen," 263, 283), who found that formaldehyde combines with phenols in the presence of hydrochloric acid, and Emil Nickel (*Zeit. f. anal. Chem.*, 1889, p. 249) states that the phenols are useful reagents for aldehydes, and *vice versa*, but as far as I am aware, the appearance of the red coloration with formaldehyde has not been previously noticed.

The precipitate obtained by sulphuric acid, formaldehyde, and phenol is so insoluble that it might well be utilized for the determination of the strength of dilute formalin solutions. When a trace of the precipitate is taken, a few drops of bromine water or iodine solution added, and then a little sodium hydrate solution, a strong violet-red coloration and a precipitate are obtained.

The silver reaction for aldehydes altogether fails with the faint traces of formalin which are readily recognisable with the milk-sulphuric and the phenol-sulphuric tests.

Moreover, all distillates from pure milk show a very faint browning with ammoniacal silver solution.

The diphenyl reaction referred to by Messrs. Richmond and Boseley is far less sensitive than the two reactions described in this paper.

DISCUSSION.

The PRESIDENT said that most of the tests hitherto employed for the detection of formaldehyde had been of such a character as to be applicable only to aldehydes as a class. The reaction shown by Mr. Hehner was, however, a very characteristic one, and likely to prove extremely useful. He had found that an aqueous solution of milk sugar gave a reaction with Schiff's reagent, and therefore distillation was necessary.

Dr. RIDEAL remarked that allusion had been made to a paper published by himself on the subject of formalin, but he thought that was hardly the right way to put it. It would be remembered that in the course of the previous year Mr. Bevan had promised a paper on the use of formalin for preserving milk samples, which he was unable to bring forward on the date for which it was announced. He (Dr. Rideal) happened to be present at the meeting on that date, and had ventured to make a few remarks regarding formalin and its detection, which the President suggested should be published, in the form of a note, in the ANALYST. Seeing that he had not attended the meeting with the intention of reading a paper, the appearance of the note in question was only due to the kindness of the meeting and the suggestion of the President; but, in the event of any question arising as to the use of Schiff's test in connection with formalin, it was rather unfortunate that this short note should have been placed after the papers of Mr. Bevan and Messrs. Richmond and Boseley, inasmuch as it was read at a previous meeting to that at which those papers were presented.

With regard to Mr. Richmond's modification of Schiff's test, it was necessary to remember that formaldehyde was the only volatile aldehyde which had to be looked for, and that the original method of applying Schiff's reagent to the distillate tested was not merely for the detection of the presence of an aldehyde, but for that of a volatile aldehyde. It seemed, therefore, to be preferable to adhere to the method suggested by himself, rather than to follow one which involved the application of the reagent to the original milk without distillation.

The colour-test mentioned by Mr. Hehner seemed to be especially useful, for the reason that it was manifested when the sulphuric acid was added to the milk in sorting samples by the machine.

Mr. WHITE observed that in the case of milk containing formalin, the clot formed on the addition of hydrochloric acid to the milk was much more difficult to dissolve than when no formalin had been added.

Mr. W. NORTHFIELD YARROW remarked that the clot referred to by Mr. White also took a yellowish tinge, if much formalin were present, while it remained whitish if there was none.

Mr. C. A. MITCHELL mentioned two tests which he had found useful for detecting

formaldehyde. One was the reduction of chromates by aldehydes, and the subsequent formation of a violet modification of the chromic salt. A mixture of 1 c.c. of nitric acid and an equal quantity of potassium chromate solution added to the distillate from the liquid under examination, produced, after a shorter or longer time, according to the quantity of the aldehyde present, a violet coloration. The other test was dependent upon the action of formaldehyde on Nessler's solution. A very small trace of formaldehyde was sufficient to produce a yellow colour, which gradually darkened and produced a precipitate at first resembling ferric oxide, but finally becoming dark gray. Ordinary aldehyde did not give this reaction.

Dr. RIDEAL remarked that the test of smell, which had not been mentioned either by Mr. Richmond or by Mr. Hehner, was very useful in detecting the presence of formalin. In milk, 1 part of formaldehyde in 25,000 could easily be recognised by its odour on warming the milk.

In the process of distillation, preliminary to the application of Schiff's test, the first portions of the distillate possessed a pronounced odour of formalin.

Mr. RICHMOND said that the fact of the non-occurrence of the blue reaction with sulphuric acid when formaldehyde was present in large quantity was mentioned in the paper by himself and Mr. Boseley (*ANALYST*, xx. 155), and the insolubility of the casein referred to by Mr. White had been noticed by Mr. Seyler.

The President had said that, under some conditions, milk sugar gave a reaction with Schiff's test, but he (Mr. Richmond) had never been able to observe such a reaction, and would like to hear under what conditions it was obtained.

Dr. Rideal had claimed priority with regard to the use of Schiff's test. As far as he (Mr. Richmond) and his colleague were concerned, they freely admitted the justice of Dr. Rideal's claim; in fact, so far from making any claim for its use, they were the first to point out its unreliability.

With regard to Dr. Rideal's remarks as to the modification of Schiff's test which they had suggested, he would like to point out that acetaldehyde was volatile as well as formaldehyde, and that its antiseptic properties (although inferior to those of formaldehyde), were sufficient to admit of its use as a milk preservative.

Mr. HEHNER said it seemed to him ridiculous to talk of priority at all in connection with Schiff's test. Long before formaldehyde came into use as a preservative, it was known that it gave a reaction with Schiff's reagent.

With regard to the test of smell, no doubt the nose was a very useful organ, but it required backing up by something more definite. Such a test was not, as a rule, accepted by chemists; in legal cases especially something in addition was required which was discernible to the eye.

The PRESIDENT said it was very difficult to define the conditions under which Schiff's test did or did not succeed, and therefore, although he had observed the coloration with milk sugar, he could not exactly lay down the conditions under which it would appear. It might be due to a small quantity of alkali.

OFFICIAL METHODS FOR THE ANALYSIS OF FERTILIZERS, ISSUED
BY THE GERMAN MANURE MANUFACTURERS' ASSOCIATION,
HARZBURG, MAY 28, 1895.

CONTRIBUTED BY H. H. B. SHEPHERD, ANGLO-CONTINENTAL GUANO WORKS, LONDON.

I. DETERMINATION OF WATER.

For the determination of moisture in superphosphates, raw phosphates, sulphate of ammonia, saltpetre, bone-meal, dried blood, horn-powder, and similar substances, 10 grammes to be dried at 100° C. until a constant weight is obtained; substances containing gypsum to be dried for three hours. Should any volatile matter, such as volatile ammoniacal salts, be present, it is to be separately determined, and the amount deducted from the loss first obtained.

II. DETERMINATION OF INSOLUBLE MATTER.

The determination of the insoluble matter, like that of the moisture, is required principally for such purposes as calculating results into the pure or dried state. It is to be carried out as follows: Ten grammes to be taken, and (a) if the solution is made in acid, to be evaporated to dryness, to render the silica insoluble, and the residue filtered off, washed, and ignited.

(b) If the solution is made in water, the residue to be filtered off, washed, and dried at 100° C. until the weight remains constant.

III. DETERMINATION OF PHOSPHORIC ACID.

A. *Retrospect*.—Owing to differences frequently occurring between the results obtained by works' chemists and the chemists of the experimental stations, it was thought desirable in the year 1889 to institute an inquiry into the matter. A meeting of the chemists of the German experimental stations was held at Bremen in 1890, in conjunction with representatives of the German Manure Manufacturers' Association, and it was decided to send uniformly mixed samples of various superphosphates to different experimental stations and works' laboratories for the purpose of comparing the molybdate and citrate methods for determination of phosphoric acid, and of testing the various processes in use for the extraction of soluble phosphate. This plan was duly carried out; but the results, especially those obtained by the molybdate method, were not considered satisfactory, and it was decided at the next meeting, held at Halle in 1891, to pursue the inquiry further. It was desired, in the first place, to make a study of the molybdate method, by employing a solution of phosphoric acid of known strength, with the view of issuing reliable working instructions for this method, and at the same time to investigate the alternative citrate method. As regards the method of extraction, a complete agreement had already been arrived at.

These further experiments were carried out simultaneously in May, 1892, by the chemists of thirteen experimental stations and five works. The results by the molybdate method were again discordant; but the citrate method gave sufficiently good results, even though the ammonium magnesium phosphate precipitate contained

some calcium phosphate. It was, therefore, decided to accept this method for the determination of the soluble phosphoric acid in superphosphates.

The cause of the differences in the results obtained by the use of the molybdate method was the subject of discussion at the fifth general meeting of the chemists of the experimental stations, held at Berlin in 1892, in which representatives chosen by the German Manure Manufacturers' Association took part, and was traced to differences in procedure between the different chemists. It was then thought that concordant results might be obtained by issuing exact directions for working the method, and by operating upon a solution of phosphoric acid of known strength and of a composition similar to a solution of superphosphate.

It transpired in the course of the discussion that three principal modifications of the molybdate method were followed—viz., that in use at the laboratory at Halle, that of Fresenius, and that of Professor Wagner of Darmstadt.

Detailed descriptions of these three processes were accordingly prepared and sent to the members, and in the spring of 1893 they were tried at five works and twenty-nine experimental stations, the results being published at the sixth general meeting held at Würzburg in the autumn of the same year. The results, however, were again disappointing, and it seemed that the more accurately the details were carried out, the greater were the divergencies experienced. It was, nevertheless, resolved to follow up the experience already gained by making still further experiments, and especially to give attention to the researches of Neubauer on the subject.

As was made known through the medium of the official report of the Dresden meeting of September, 1894,* this resolution was acted upon by the chemists of the experimental stations, but without the co-operation of the committee of the Manure Manufacturers' Association. A solution of absolutely pure potassium phosphate was prepared by neutralizing pure potassium carbonate with phosphoric acid obtained by the combustion of pure phosphorus, and with this solution fresh experiments were made both with the molybdate and the citrate methods. No conditions, however, were laid down for working the molybdate method, it being left to each chemist to use any recognised mode of procedure according to his judgment. The results obtained were most satisfactory, as, indeed, would be expected when dealing with a chemically pure substance. Operating upon 0.150 gramme phosphoric acid for each experiment, nineteen out of twenty-three of the molybdate determinations came within 0.001 gramme, corresponding to .1 per cent. in a superphosphate containing 15 per cent. phosphoric acid. The determinations by the citrate method showed also a remarkable agreement, the results of ten chemists not deviating more than 0.001 to 0.002 gramme from the mean.

As a consequence, it was decided to recommend that official analyses should continue to be carried out by some approved modification of the molybdate method.

The matter, therefore, stands at present thus, that for ordinary superphosphate testing the citrate method may be used, but for official analyses the molybdate method is to be employed. As, however, complete agreement on matters of detail was not arrived at, it was left to each chemist to adopt such plan as according to his judgment afforded the most accurate result. The analytical commission of the

* *Landwirthsch Versuchsstat.*, xlv., 390.

German Manure Manufacturers' Association, on the strength of the experience obtained during the foregoing investigation, recommends the so-called Halle method, a description of which follows, as the one which furnished, comparatively speaking, the most concordant results.

B. *Analysis of Superphosphates and Raw Materials.*—Agreement has been arrived at upon all points here referred to.

1. *Preparation of the Samples.**—(a) Dry samples of phosphates or other artificial fertilizers may be sifted and then mixed.

(b) In the case of damp materials, where this is not possible, the preparation is to be confined to a careful mixing by hand.

(c) In raw phosphates and animal charcoal, for reasons before mentioned, the moisture is first to be determined.

(d) In dealing with substances which lose water in the process of reducing to powder, the moisture is to be determined in the coarse as well as in the fine, and the results of the analysis calculated into the original state as received.

2. *Extraction of Superphosphate.*—The extraction of superphosphate is to be carried out as follows: Twenty grammes superphosphate to be introduced into a litre flask with 800 c.c. water and shaken continuously for half an hour, then made up to the mark with water, mixed, and filtered. The filtration is to be taken in hand promptly, as if left long standing, phosphoric acid separates out.

For carrying out the shaking operation, shaking machines are desirable. They can be worked either by hand or by motor, and should make about 150 revolutions per minute.

Solutions of concentrated superphosphate (so-called double superphosphate) should be boiled with nitric acid before precipitating the phosphoric acid, to convert any pyrophosphoric acid present into orthophosphoric acid. Ten c.c. of nitric acid of 1.4 specific gravity are sufficient for 25 c.c. solution. For ordinary work the citrate method may be used, but for official analyses the molybdate method is to be employed.

For the determination of the phosphoric acid in bone-meal, fish guano, meat-meal and raw phosphates,† and the total phosphoric acid in superphosphates, 5 grammes are to be dissolved in 50 c.c. nitrohydrochloric acid, consisting of 3 parts hydrochloric acid of 1.2 specific gravity, and 1 part nitric acid of 1.25 specific gravity; or the substance may be boiled for half an hour in a mixture of 20 c.c. nitric acid of 1.42 specific gravity and 50 c.c. sulphuric acid of 1.8 specific gravity. After cooling, the solution is to be made up to 250 c.c.

(To be continued.)

* In accordance with the resolution of the third general meeting of the chemists of the experimental stations held at Bremen in 1890.

† Thomas-meal is referred to in another section.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD ANALYSIS.

Sterilization of Milk. Prof. Flugge. (*Rev. Inter. de Bibliographie*; through *Rev. Inter. des Falsif.*, ix., pp. 29, 30.)—While the pathogenic microbes of tuberculosis, cholera, diphtheria, and typhoid fever are killed by boiling milk for a short time, such is not the case with the microbes of infantile diarrhœa. These latter multiply at a comparatively high temperature, hence infantile mortality from diarrhœa is higher in summer. Soxhlet's method of milk sterilization by heating for three-quarters of an hour is ineffectual.

Flugge has cultivated the following bacteria from milk which had been heated to 95° C.: four anaerobic bacilli, of which the most common is *Bacillus butyricus* of Bothin; and twelve aerobic bacilli. The latter, like the former, have spores which resist boiling for two hours at 100° C. They curdle milk visibly in from twelve to fourteen days, but the milk contains millions of bacilli before any alteration is evident. The aerobic organisms form peptones which cause diarrhœa. Flugge concludes that the sterilized milks of commerce are dangerous preparations, the sale of which ought to be stopped by the public authorities. As a means of preventing the growth of peptonizing bacteria, he further recommends that milk should be boiled for six minutes, and then cooled below 18° C., and consumed within twelve hours. After sterilizing, milk should be kept in a closed vessel, hermetic sealing being useless.

W. P. S.

Copper in Wine. H. Karsten. (*Zeits. österr. Apoth. Ver.*, 1896, xxxiv., 84; through *Chem. Zeit. Rep.*, 1896, 37.)—A number of cases of vomiting and diarrhœa have recently occurred in Switzerland, which seemed probably due to copper poisoning. A bright steel blade immersed in the suspected wine gave a distinct copper reaction in twelve hours, the metal evidently gaining access to the wine owing to the unripe grapes having been sprayed with a preparation of copper in the vineyards.

F. H. L.

ORGANIC ANALYSIS.

Reactions of Formaldehyde. Romijn. (*Nederl. Tijdschr. voor Pharm. Chem. en Tox. juïn*, 1895; through *Rev. Inter. des Falsif.*, ix., pp. 13, 14.)—The author converts formaldehyde into hexamethylene-tetramine by ammonia. This body forms regular crystals, and gives well-defined reactions. The substance under examination is distilled with water, and a drop of the distillate mixed on a microscope-slide with a drop of ammonia; after evaporation the crystalline residue is examined, and tested with the following reagents:

1. *Mercuric Chloride in excess.*—Formation of crystals; hexahedral at once, octahedral after a time. The latter are easily obtained from a solution containing 1 : 10,000, and are still visible in 1 : 100,000.

2. *Platinic Chloride*.—Octahedral crystals, perceptible in 1 : 1,000, but not in 1 : 10,000.

3. *Phosphomolybdic Acid*.—Rhomboidal crystals from solutions of 1 : 10,000.

4. *Potassium Iodide or Bismuth Iodide in Potassium Iodide*.—In slightly acid solutions yellow crystals are formed, which are plainly seen in solutions containing 1 : 1,000, also in 1 : 10,000 after a time.

5. *Hydrochloric Acid Solution of Stannous Chloride*.—Needle-like crystals from a solution containing 1 : 1,000.

6. *Mercuric Iodide in Potassium Iodide, slightly acidified with Hydrochloric Acid*.—Bright yellow hexagonal crystals from solutions containing 1 : 10,000.

7. *Iodine in Potassium Iodide*.—Rhombic crystals easily seen in solutions of 1 : 1,000.

8. *Picric Acid*.—Crystals from a solution of 1 : 1,000.

Many other bodies, as silver nitrate, mercurous nitrate, gold chloride, glacial acetic acid, etc., also give crystalline bodies, but the reaction with mercuric chloride is chiefly relied upon.

W. P. S.

A New Method of analysing Alkaline Benzoates. G. Rebière. (*Journ. Pharm. et Chim.*, 1896, 113-116.)—This is based on the fact that most mineral acids liberate benzoic acid from its metallic compounds. A small quantity, p , of the benzoate is treated with a sufficient quantity of hydrochloric acid, and the mixture evaporated to dryness. The benzoic acid and the excess of hydrochloric acid are volatilized, and the metal is left as chloride. The amount of chlorine is then determined with decinormal silver nitrate solution, n c.c. being taken.

To determine the benzoic acid, the same weight p of the benzoate is dissolved in 50 to 60 c.c. of water, and n c.c. of decinormal sulphuric acid added. This exactly saturates the base, and the liberated benzoic acid is titrated with decinormal soda, phenol-phthalein being used as indicator.

This method can be used with most metallic benzoates, but in the case of lithium benzoate care must be taken to add the smallest possible excess of hydrochloric acid, and to evaporate on the water-bath in order to avoid loss by volatilization. In the case of ammonium benzoate the process is modified as follows: A given weight, p (about 0.20 gramme), of the benzoate is dissolved in 20 c.c. of decinormal soda, and the mixture boiled until all ammonia is expelled. The excess of alkali is then determined with decinormal acid (n c.c.). The amount of ammonium will be $(20 - n) \times 0.0018$. It is necessary to prove that the benzoate itself does not contain free acid. For the estimation of the benzoic acid, the same weight, p , of ammonium benzoate is dissolved in 20 c.c. of water, and $(20 - n)$ c.c. of decinormal sulphuric or hydrochloric acid added. The liberated acid is then titrated with decinormal soda.

C. A. M.

The Biuret Reaction. Hugo Schiff. (*Berl. Ber.*, xxix., 298.)—It is shown that not only biuret and albuminous substances give a red coloration with cupric sulphate and caustic soda, but all bodies which contain two CONH_2 groups, com-

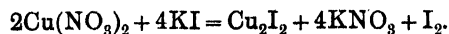
bined in the molecule with a single carbon or nitrogen atom, or joined by one or more CONH groups. Oxamide also gives the reaction. O. H.

A Method of Estimating Uric Acid. E. Riegler. (*Zeit. anal. Chem.*, 1896, 31-34.)—This method is based on the property of uric acid of reducing Fehling's solution. On the assumption that one molecule of uric acid reduces two molecules of copper sulphate, one gramme of uric acid should correspond to 0.7556 gramme of copper. In the actual determinations made by the author using pure uric acid the ratio was higher than this, one gramme of uric acid giving 0.8000 gramme of copper as the mean of ten determinations.

For the determination of uric acid in urine, it is necessary to first separate it in a form in which it can be used with Fehling's solution. This may be effected by the method of Ludwig-Salkowski (*Zeit. anal. Chem.*, xxiv., 637) or more rapidly by that of Fokker (*ibid.*, xiv. 206). Two hundred c.c. of urine are mixed with 10 c.c. of a saturated solution of sodium carbonate, allowed to stand for half an hour, and filtered from the precipitated phosphates. The precipitate is washed with 50 c.c. of hot water, and to the filtrate and wash-water 20 c.c. of a saturated solution of ammonium chloride added. The liquid is well stirred, and after five hours filtered, preferably through a Schleicher and Schüll filter, No. 597, 11 cm. The precipitate is washed with 50 c.c. of water, and then introduced by means of a jet from a washing-bottle into a 300 c.c. beaker. Several drops of potash are added to clear the liquid, then 60 c.c. of Fehling's solution, and the whole well stirred. The beaker is then heated on a wire gauze until the liquid boils, the boiling being continued for five minutes. When the precipitate has subsided, the liquid is filtered through a small tough filter (Schleicher and Schüll, No. 590, 9 cm.), the precipitate well washed, and dissolved in 20 c.c. of nitric acid (specific gravity, 1.1), the filter being washed with 60 c.c. of water.

To this solution dry powdered sodium carbonate is added little by little until there is a permanent turbidity. The liquid is then cleared by the cautious addition of sulphuric acid, and made up to 100 c.c. Twenty-five c.c. of this are placed in a 100 c.c. flask, one gramme of potassium iodide in 10 c.c. of water added, allowed to stand for ten minutes, and titrated with thiosulphate solution (1 c.c. = 0.002 gramme uric acid), using starch as the indicator. To the total amount of uric acid found in the 200 c.c. of urine, an additional 0.030 gramme should be added to allow for the solubility of the ammonium urate in urine.

Thiosulphate solution of the right strength is readily obtained by diluting 126 c.c. of a decinormal solution to 500 c.c. The reaction employed is :



The reduced cuprous oxide may also be weighed directly or reduced to metallic copper, as in the estimation of sugar. In the latter case the amount of copper, multiplied by the factor $\frac{1000}{0.8000} = 1.25$, gives the corresponding amount of uric acid.

C. A. M.

Detection of Vegetable and Animal Oils in Mineral Oils. C. Halphen. (*Ann. Chim. Analyt.*, 1896, 29.)—The author finds that the reaction proposed for this purpose by De la Royère, and which consists of adding to a few drops of the suspected oil, contained in a porcelain dish, two drops of a solution of fuchsine just decolorized with alkali, is valueless. Many carefully prepared mineral oils have an acid reaction, and restore the colour of the fuchsine solution. Soaps are often added to mineral lubricating oils, and in such cases the free acid of any added vegetable oil is neutralized, and consequently they do not respond to the above test.

The presence of soap in such oils may readily be detected by their action on a solution of Congo-red which has just been rendered violet by hydrochloric acid. Soaps, notwithstanding their alkaline reaction, change the colour of this indicator to red. (?)

W. J. S.

On the Determination of the Degree of Oxidation of Oils. W. Bishop. (*Journ. Pharm. et Chim.*, 1896, 55-61.)—The process of Livache, which consists in determining the increase of weight in oils when exposed to the action of oxygen in contact with finely-divided lead, is only serviceable in the case of linseed-oil. In other oils the oxidation proceeds too slowly. In order to obtain the most rapid oxidation, the main essential is to have the oil in as finely-divided state as possible by means of a porous neutral substance. The author found precipitated silica a suitable dividing agent, but the absorption was still too slow for practical purposes. The addition of manganese resinate to the silica gave the desired result. The commercial resinate is purified by treatment with ether or petroleum spirit, filtering, and evaporating the ether. The dry residue, which the author found to contain 9.80 per cent. of oxide, is powdered and kept in a stoppered bottle.

The method of determining the oxygen absorption of oils is as follows: From 5 to 10 grammes of the oil are weighed into a dish, and for 100 parts of oil exactly 2 parts of the resinate added; that is, for 10 grammes 0.2 gramme. The mixture is agitated on the water-bath until the resinate has dissolved, and then allowed to cool. One gramme of silica is weighed into a flat dish provided with a glass stirring-rod, and then drop by drop, by means of a pipette, 1.02 grammes of the resinated oil (1 gramme of oil + 0.2 gramme resinate) is added. The mass is intimately mixed, and spread over the bottom of the dish, and is left at a temperature of from 17° to 25° C. for drying oils, and of 20° to 30° C. for other oils. The dish is weighed after six hours, and twice again in the twenty-four hours, and so on until the maximum is attained, the mass being stirred up after each weighing. The maximum increase in weight, multiplied by 100, gives the degree of oxidation. The detailed results obtained with linseed, poppy, cotton seed, and earthnut oils are given in Tables I. and II.

I.

		Linseed-oil, Native. Density = 0.9357 at 15° C.		Linseed-oil, La Plata. D = 0.9304 at 15° C.		Linseed-oil, Native. Pure. D = 0.9322 at 15° C.		Containing 5 per cent. resin oil. 5 per cent. mineral oil. D = 0.9323 at 15° C.	
		T = 28°-22° C.	T = 17°-23° 17° C.	T = 28°-22° C.	T = 17°-23° 17° C.	T = 23°-17° C.	T = 17°-14° C.	T = 23°-17° C.	T = 17°-14° C.
Increase per cent. after	Hours.								
	6	12.8	4.95	11.20	3.75	13.5	1.45	11.50	1.70
	22	16.10	16.70	14.10	14.30	16.30	14.75	14.80	13.55
	24	16.60	17.40	14.30	14.90	16.40	15.25	14.90	13.95
	30	16.50	17.70	14.70	15.10	16.20	15.25	14.80	13.95
	48	15.50	17.70	15.00	14.70	15.90	15.95	14.60	14.55
	72	15.10	17.70	14.70	14.90	...	16.25		14.75
	96	14.80	17.30	14.50	14.80		
	120	15.00	...	14.30		14.80	...	13.80	
	144	14.80	...	14.10		...	16.65		15.35
216	...	17.10		14.80	...	15.85		14.45	

II.

		Poppy-oil. D = 0.9242 at 15° C.		Cottonseed-oil, Containing cotton "stearin." D = 0.924 at 15° C.		Earthnut-oil, Mozambique. D = 0.916 at 15° C.	
		T = 28°-22° C.	T = 23°-17° C.	T = 28°-22° C.	T = 23°-17° C.	T = 32°-18° 34. 5°-14° C.	T = 23°-17° C.
Hours.							
Increase per cent. after	6 ...	4.8	0.20	1.30	0.10	0.80	- 0.20
	22 ...	13.60	11.45	7.30	5.70	3.50	- 0.20
	24 ...	13.90	11.55	7.60	6.20	3.80	- 0.10
	30 ...	14.50	12.45	8.20	7.40	4.70	0.80
	48 ...	14.10	14.30	8.60	8.40	5.30	3.60
	72 ...	13.00	...	8.40	8.50	6.10	4.40
	96 ...	12.50	13.35	8.20		6.70	...
	120 ...	12.00	12.75	8.00	...	6.50	...
	144 ...	10.40	...	7.80	8.00	6.50	4.80

A general summary of results is given in the subjoined table. Earthnut-oil may be considered as intermediate between semi-drying oils like cottonseed, and non-drying oils like colza and olive. For the latter a slightly more elevated temperature

should be employed (20° to 30° C.). Even then oxidation is not complete in a short time.

III.

Oils.	Density.	Degrees of Oxidation.	Mean Degree.
Linseed, native	0.9327	17.70—16.40	17.05
„ la Plata	0.9304	15.45—15.00	15.20
Hempseed	0.9287	14.55—14.30	14.40
Poppy, native	0.924	14.50—13.90	14.20
Nut	0.924	13.70	13.70
Cotton-seed with stearin ...	0.924	8.60	8.60
„ without stearin	0.923	9.60—9.30	9.45
Sesame, Senegal	0.9215	8.95—8.50	8.70
„ Indian	0.921	7.40	7.40
Earthnut, African	0.916	6.70	6.70
„ white	0.916	6.50	6.50
Colza, native	0.9142	6.40 ?	6.40 ?
„ Indian	0.9137	5.90—5.80 ?	5.85 ?
Olive	0.9155	5.30 ?	5.30 ?

The degree of oxidation can be controlled by the Hübl number, and in the author's opinion is in many cases more complete than the latter, since it shows at once the absorption power and the rapidity of oxidation. In applying the method to the detection of vegetable oils in lard, it is preferable to employ the liquid fatty acids, for the separation of which the author recommends Halphen's process (ANALYST, xix., 282).

C. A. M.

The Bromine Heat-Value of Fats and Oils. J. A. Wilson. (*Chem. News*, 1896, 87.) — The author has examined Hehner and Mitchell's thermal process (ANALYST, xx., 146), and considers it in the main accurate and convenient. Using a test-tube packed in cotton-wool as his calorimeter, he found that with cocoanut-oil the rise of temperature multiplied by 5.5 gave at once the correct iodine value. With tallow, lard, butter, etc., it was necessary to add 1 before multiplying by that factor. This was shown in the following table :

Oil or Fat.	Rise of Temperature with Bromine.	Calculated Iodine Value.	Observed Iodine Value.
Cocoanut-oil	1.5	8.2	8.4
S.A. tallow	7.0	43.2	44.0
Olive-oil (pure)	14.0	82.5	82.0
Rape-oil	18.0	104.5	103.4

W. J. S.

The Use of the Calorimeter in Butter and Lard Analyses. E. de Schweinitz and J. A. Emery. (*Jour. Amer. Chem. Soc.*, xviii., 1896, 174-179.) — The samples of lard and butter which the authors were examining by the usual methods were prepared by washing, melting, filtering and drying at 100° C., and

sent to Professor Attwater for combustion, without any information being given except that they were fats. The results of the analyses of butters and oleomargarines are given in the following table: I.

Sample No.	Original substance.								Fat.			
	Manufacturer.	Specific Gravity at 100° C.	Water, per cent.	Vol. $\frac{1}{10}$ Ba(OH) $\frac{1}{2}$ for 2.5 grs.	Salt, per cent.	Casein, per cent.	Ash, per cent.	Melting-point, ° C.	Iodine No.	Combustion Calories, per gr. Attwater.	Cottonseed-oil. Becchi's Test.	Colouring Matter.
9	Oakdale Manufacturing Co., oleomargarine	0.8835	8.53	0.42	5.17	1.36	5.95	23.0°	52.80	9.599	Purple brown	
12	Oakdale Manufacturing Co., oleomargarine	0.8906	8.00	0.35	3.75	2.59	—	25.0°	54.49	9.620	Purple brown	
14	Vermont Manufacturing Co., oleomargarine	0.8828	9.47	0.35	5.31	1.33	5.67	25.0°	66.50	9.795	Purple brown	
16	Vermont Manufacturing Co., oleomargarine	0.8874	9.00	0.30	3.68	1.66	3.68	22.5°	66.59	9.649	Purple brown	
18	Swift & Co., oleomargarine	0.8830	8.32	0.22	2.12	0.81	2.19	24.0°	60.67	9.644	No change	Highly coloured
19	Hammond Manufacturing Co., oleomargarine	0.8891	9.15	0.22	6.69	1.43	6.21	25.0°	60.53	9.607	No change	Very highly coloured
22	Woodlawn oleomargarine	0.8880	9.23	0.35	3.80	1.52	3.68	22.5°	61.80	9.670	No reaction	Highly coloured
26	Elgin Creamery Butter	0.8925	8.32	11.10	3.81	1.27	3.64	35.3°	37.75	9.327		
27	Plains Va. Butter	0.8979	11.43	8.55	4.05	1.84	4.93	36.1°	36.86	9.362		
28	Armour & Co.'s best butter	0.8984	12.98	10.82	4.04	1.30	3.94	33.5°	41.20	9.320		
29	Armour & Co.'s oleomargarine	—	—	—	—	—	—	—	49.91	9.601	No reaction	

The figures obtained for butter-fat differ slightly from those given by Stohman, who found the heat equivalent, as determined by the potassium chlorate method, to be 9.192 small calories per gramme, while by the oxygen method it was 9.231 calories.

The increase in the calories of mixtures of butter and oleomargarine was in proportion to the amount of the latter added. In Table II. the results were obtained by mixing Elgin butter (26) with Woodlawn oleomargarine (22).

II.

Sample.	Actual I. No.	Theoretical I. No.	Actual Combustion Calories, per gramme.	Theoretical Combustion Calories, per gramme.
$\frac{3}{4}$ E. and $\frac{1}{4}$ W. ...	43.90	43.76	9.391	9.412
$\frac{2}{4}$ E. and $\frac{2}{4}$ W. ...	48.01	49.77	9.416	9.498
$\frac{1}{4}$ E. and $\frac{3}{4}$ W. ...	54.40	55.78	9.491	9.584

In the case of lards taken from different sources, the results are not so distinctive, but, taken in conjunction with the iodine absorption, will be of use.

III.

ANALYSIS OF LARDS FROM ARMOUR AND CO.

Quality.	Melting-point.	Iodine No.	Combustion Calories, per gramme.	Cottonseed-oil, Becchi's Test.
Lard, leaf	—	56·85	9·621	None.
„ caul fat	40·0°	58·61	9·573	Slightly darker.
„ intestinal fat	40·7°	54·74	9·581	„
„ head	29·5°	68·79	9·503	None.
„ trimmings	—	65·57	9·606	„
„ mixture of all fats	—	63·86	9·654	„
„ compound, 1st grade .	—	86·18	9·583	Purple brown.
„ „ 2nd grade	—	86·57	9·530	„
„ shield	—	61·01	9·598	None.
„ special pure	37·5°	63·63	9·617	„

C. A. M.

INORGANIC ANALYSIS.

On the Estimation of Water in Silicates by the Borax Method. P. Jannasch and P. Weingarten. (*Zeit. Anorg. Chem.*, xi., 37.)—This is a modification of the process already described (*ibid.*, viii., 352), which was designed to replace the method of water estimation where the mineral is opened out with plumbic oxide, and which was found to yield irregular results in the case of silicates rich in calcium. The new process permits of the portion of material operated upon being used for other subsequent determinations.

1·5 grammes of borax, dried as described below, are placed in a platinum boat 7·5 c.m. long by 1·5 deep, which is put into the middle of a thin-walled combustion tube 75 c.m. in length, followed by a plug of glass wool. To free the tube from the last traces of moisture it is heated gently in a combustion furnace for half an hour while a current of dry air is passed through, great care being taken to avoid fusing the borax. After this the tube is allowed to cool, the air current is stopped, and the weighed quantity of the substance under examination is mixed as rapidly and as intimately as possible with the borax in the boat by means of a spiral of platinum wire. The boat is pushed back to its former position, the glass-wool plug replaced, and the tube again connected to the air supply. The unweighed calcium chloride tube at the exit end of the combustion tube is changed for a weighed one, to the outer end of which the first tube is attached.

The part of the tube containing the boat is then very gradually heated up, the remainder being kept as cool as possible by not covering it with tiles.

When the borax is fused to a clear homogeneous glass in which bubbles are no longer being formed, the air-current is started once more, and the whole length of the tube is heated with a Bunsen burner to drive the moisture into the calcium chloride tube, which, after cooling to the temperature of the balance-room, is reweighed.

Should fluorine be present in the substance analysed, a layer of granulated lead chromate 5 c.m. long, with a glass wool-plug at either end, is placed between the platinum boat and the exit end of the combustion tube, to absorb any volatile fluorides.

The powdered borax employed in the process is prepared (*Zeit. Anorg. Chem.*, viii., 352) by heating pure crystallized borax to incipient fusion in a platinum dish or crucible, allowing to cool, reducing to fine powder and finally heating to a dull red heat while the powder is stirred with a thick platinum wire.

C. H. C.

Volumetric Determination of Copper. **M. Rupeau.** (*Bull. Soc. Pharm. de Bordeaux*, October, 1895; through *Ann. Chim. Analyt.*, i., 32.)—The author proposes the employment of a solution of picric acid, which gives with copper a precipitate insoluble in ammoniacal liquids. The standard picric acid solution is made by dissolving rather more than 7.5 grammes of the acid in hot water, and, after adding ammonia (30 to 40 c.c.) till a decided odour is manifested, making up to 1 litre. After standing for some time, the solution, is filtered and standardized against a solution of 1 gramme of pure red copper, dissolved in nitric acid and made up to 100 c.c. with water (1 c.c. = 10 mgms. Cu). The picric acid is then run in, with constant agitation. So long as a green tinge is observed there is no danger of overstepping the end point; but when this is approached the precipitate is allowed to settle, and the addition is proceeded with cautiously until a decided yellow tinge without any tendency to green is obtained in the clear supernatant layer of fluid. The titration of the solution under examination is performed in an exactly similar manner.

When silver is present, 10 c.c. of ammonia must be added to the nitric solution to keep this metal in solution while titrating, and the same holds good for zinc as well. Lead behaves differently. The oxide thrown down by the ammonia obscures the final reaction, but this may be obviated by adding a little tartaric acid. Iron is removed by simply boiling the solution, adding ammonia to precipitate the ferric oxide. In the most complex case likely to occur—*i.e.*, one in which the copper is associated with all the four metals mentioned above—the method is as simple as when iron is the only admixture.

C. S.

A Critical Examination of the Processes for the Estimation of Phosphoric Acid. **C. Meineke.** (*Chem. Zeit.*, 1896, xx., 108.)—The author has already stated, some ten years ago, that it is perfectly possible by gentle ignition to convert the yellow ammonium phospho-molybdate into a form in which it can safely be weighed with the assurance of its containing a definite percentage of phosphorus. He has now reinvestigated his process, checking it on specimens of phosphorite, on pure disodium phosphate, and on silver phosphate, and he finds that the phosphomolybdic anhydride so obtained contains constantly 3.949 (± 0.008) per cent. of P_2O_5 ; corresponding, therefore, to the formula $24MoO_3 + P_2O_5$, which (taking $Mo = 95.9$, $P = 30.96$, and $O = 15.96$) demands 3.944 per cent.

The molybdate solution should be prepared by dissolving 150 grammes of the ammonium salt in 150 c.c. of ammonia (specific gravity 0.91) and 850 c.c. of water, pouring the whole into 1 litre of nitric acid (specific gravity 1.2). The liquid is heated to 90° C. for ten minutes, filtered from the molybdic acid, and preserved in the dark.

In testing solutions poor in iron, the precipitation is so arranged that at least 5 per cent. of ammonium nitrate is present. To ensure this, ammonia (specific gravity 0.91) and nitric acid (specific gravity 1.4) are prepared, which, when mixed together in equal volumes, yield a moderately acid solution of the nitrate of about 53 per cent. concentration. These are added in the necessary amounts in succession to the liquid under examination, and when the temperature has fallen to 50° C., the molybdate is added, the whole thoroughly stirred, and set aside till clear. If the solution, on the other hand, contains much iron, at least 10 per cent. of ammonium nitrate must be present, and enough free nitric acid to represent 5 to 10 per cent. of the 1.4 strength. The solution is heated to the boil, the molybdate introduced, stirred up, and set aside. Further heating after the addition of the precipitant must be avoided. The precipitate should be pale yellow, and the liquid must also be light in colour.

It is washed with a liquid consisting of 100 c.c. each of the same acid and ammonia diluted to 1 litre with water. A final rinse in pure cold water is given, followed, if desired, by alcohol and ether, and the filter is then dried. It is ignited in a porcelain basin or crucible over an argand, or in a muffle, at a temperature which is not visibly red by daylight. When hot, the residue has a somewhat grayish colour, but when cold it is blue-black. It must be free from yellow or green particles, and also from free molybdic acid; and it should be cooled in the desiccator.

The author has also examined the statement that the presence of ammonium chloride in solutions containing much iron, which are to be precipitated by molybdate solution, is to be avoided. A series of careful experiments show that this substance, even when present in abnormally large amounts, is entirely without action on the results.

The remainder of the paper contains a long and elaborate examination of the two processes—Maercker's and Wagner's—for the estimation of phosphoric acid by magnesia mixture when following the molybdate precipitation. The former neutralises the ammoniacal solution of the yellow precipitate, introduces the magnesia mixture, and finally adds ammonia in $2\frac{1}{2}$ or 3 per cent. excess. Wagner prefers to add the reagent direct to the alkaline solution, which should contain the same amount of free ammonia. Both processes are liable to several errors: the precipitate is somewhat soluble, it is apt to vary in composition, and, according to the temperature of ignition, greater or less loss of phosphoric acid may occur. Neubauer has published a table of corrections for this last source of error, but it cannot be considered exact. The present author, when using Wagner's process, prefers to ignite the precipitate gently, weigh it, ignite again over the blow-pipe to constant weight (10 to 15 minutes) and weigh once more. The original precipitate, consisting of an indefinite mixture of pyro- and meta-phosphate, suffers a loss of P_2O_5 corresponding to the $Mg(PO_3)_2$ it contains; and if this loss be multiplied by 2.56, and the weight so obtained be subtracted from the original, the remainder expresses the true amount of pyrophosphate in the compound. In this manner the quantity of phosphoric acid in the precipitate can be determined exactly: the yield usually being a trifle above the theoretical. It may be observed that without correction, Wagner's process gives sufficiently exact results for many practical purposes, such as the analysis of manures: for when 1 gramme of substance is taken, the phosphoric acid comes out only about 0.2 per cent. too low.

With Maercker's modification, the original precipitate is apt to vary in composition still more; but good results are to be obtained by preventing the precipitation of the magnesium phosphate from occurring too early during the addition of the reagent. Experiments show that it is better to effect this by means of ammonium citrate than by a large excess of hydrochloric acid; and the process is best arranged so that the liquid remains clear until half of the necessary amount of magnesia mixture has been run in. In all cases the precipitate must be ignited strongly to constant weight, in order to remove the molybdic acid which is always present.

F. H. L.

REVIEW.

PRACTICAL STUDIES IN FERMENTATION, BEING CONTRIBUTIONS TO THE LIFE HISTORY OF MICRO-ORGANISMS. By EMIL CHR. HANSEN, Ph.D. (London: E. and F. N. Spon.) Price 12s. 6d.

This work, which is a translation of the last edition of Dr. Hansen's widely-known "*Untersuchungen aus der Praxis der Gärungsindustrie*," together with some additions, presents, for the first time in the English language, a collective *résumé* of the author's work in connection with the morphology and physiology of the fermentation fungi, and will in this form doubtlessly be welcomed by many who have been hitherto unable to peruse it in the original.

It contains a summary of Dr. Hansen's investigations into the life history of the fungoid organisms which play such an important part in the great fermentation industries, especially in those devoted to the production of beer. Dr. Hansen discovered some twelve years since that differences and distinctions, quite as pronounced as in the more highly-organized plants, were to be found in these lowly organisms, and that some of the species were able to exert quite as baleful an influence on the fermentations as the bacterial organisms. This discovery led to Dr. Hansen's great and important reform, which consisted in the introduction into fermentation operations of yeasts derived from a single cell, and therefore consisting of one species or variety only. This principle has proved itself of enormous value to all the fermentation industries; and though it was originally applied only to those engaged in the production of beer, has been extended to other branches, such as distilling, wine and cider making, the manufacture of pressed yeast, etc., and even to those, such as the manufacture of cheese and of tobacco, in which fermentations caused by bacterial organisms play an important part.

Details of the various operations required for the isolation of the single yeast cell, and of cultivating from it yeast in quantities sufficiently large for practical application, are given, together with much information of a highly useful nature to the practical brewer. In addition to this, much will be found which is of great interest to the pure scientist, not the least being the account of the author's experiments, by means of which he has succeeded in effecting artificial variations of a more or less permanent nature in a number of the yeast fungi. Altogether, the work is one of the most remarkable of modern times, and is of such vast importance, that no one engaged in any of the fermentation industries can well afford to be without it.

The task of translation, which has been entrusted to Dr. A. K. Miller, has been excellently performed.

W. J. S.

THE ANALYST.

MAY, 1896.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday, April 1, 1896, at 8 p.m., in the Chemical Society's Rooms, Burlington House, the President (Dr. Stevenson) occupying the chair. The minutes of the previous meeting were read and confirmed.

The following gentlemen were elected: As members—F. W. Richardson and Walter Thorp, B.Sc. (Lond.).

Mr. Rhys Pendrill Charles, F.I.C., F.C.S., Public Analyst for the County of Brecon and the Boroughs of Carmarthen and Neath, was proposed for election as member.

The following papers were read: "The Bacteriological Examination of Water for the Typhoid Bacillus," by T. H. Pearmain and C. G. Moor, M.A.; "Note on the Estimation of Formic Aldehyde," by Harry M. Smith.

The PRESIDENT, in referring to the discussion which had been announced on the subject of the form of certificates, as affected by the recent judgment in the case of *Fortune v. Hanson*, said that a verbatim copy of this judgment had been obtained, which some members of the Society wished should be read. The Council had ordered the issue of a circular embodying the judgment, and made certain recommendations. He (the President) thought that if the judgment were right, and members would consider the circular and the suggestions which it contained, it might perhaps be better to adjourn any discussion to some future meeting, if such discussion was found to be necessary.

Dr. DYER (Hon. Secretary) then read the notes of the judgment of Mr. Justice Hawkins and Mr. Justice Kennedy in the case of *Fortune v. Hanson*, from "The Law Reports," 1896, March 2 (vol. i., Q.B., part iii., p. 205), and also the forms of certificate suggested by the Council for the cases of watered milk, and butter containing excess of water, the Council being of opinion that the decision of the court should be regarded as applying only to watered milk and the analogous case of watered butter.

The Council had decided to recommend the following forms of certificates:

I.—WATERED MILK.

"I am of opinion that the said sample contains the parts as under:

Milk
Added water

100-00

"This opinion is based upon the fact that the sample contained only.....per cent. of non-fatty solids, whereas normal milk contains at least 8.5 per cent. of non-fatty solids."

II.—WATERED BUTTER.

"I am of opinion that the said sample contained the parts as under :

Water	per cent.
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"Genuine butter should not contain more than 16 per cent. of water."

He (Dr. Dyer) observed that Mr. Justice Hawkins had said that the magistrates ought to be informed on the certificate as to the total percentage of water found in the sample. The Council was of opinion—and he thought that anybody carefully reading the judgment would be of opinion—that that might possibly be construed as a figure of speech. What the judge substantially laid down was that the magistrates should have before them the result of the analysis, and the standard upon which the adulteration was calculated. No doubt the judge thought—as a layman would be likely to think—that the water itself was the standard, and therefore he mentioned water. If he had known that the calculation was made upon the non-fatty solids, he would probably have referred to non-fatty solids. The Council felt that the forms of certificate suggested complied absolutely with the spirit of the judgment in question.

The circular, while recommending public analysts to adopt the forms of certificate suggested, also recommended them to submit these forms of certificate to the clerks of their local authorities, as coming from the Council of the Society of Public Analysts.

Mr. RAYMOND ROSS asked whether it would not be well, in the case of the certificate relating to watered milk, to add the words "not less than" and "not more than" to the figures?

Dr. DYER said it seemed to him that this might introduce a little complication, although undoubtedly it would be a more accurate statement of the analyst's opinion.

Mr. ROSS : And perhaps would prevent any dispute as to whether the results of two analysts absolutely and exactly coincided.

Dr. DYER observed that such a qualification was virtually implied further on, in the words, "whereas normal milk contains *at least*." This point had not been overlooked, but had been very carefully considered by the Council.

Mr. A. E. EKINS said that for some time past he had been in the habit of putting the figures of the analyses of milks in the corner of the certificate. Several defendants had been represented by solicitors, but in no case had they been able to upset the certificate, and he thought the practice could not be regarded as incorrect.

The PRESIDENT said he had been in the habit of acting as Mr. Ekens had done, but this did not appear to completely conform to the requirements of the judgment, and the Council had thought it best to frame a certificate or certificates which, so far as they could ascertain, were in accordance with those requirements; and they recommended that every analyst should submit such certificates to the legal advisers of his authority, each analyst being guided, of course, by the opinion of those who advised him in legal matters. More than this the Council could not do.

Mr. CASSAL said he had great pleasure in rising to propose that the Society should pass a very cordial vote of thanks to their past President, Mr. Hehner, for the admirable evidence which he had recently given before the Food Products Adulteration Committee of the House of Commons on behalf of the Society of Public Analysts (applause). It would be within the recollection of the Society that a strong desire had been expressed that several of the active members of the Society should give evidence before this Committee, and that the Council had appointed three members—Mr. Hehner, Mr. Allen, and himself—to represent the Society. For reasons which at present were not very clear or satisfactory, the chairman of the Committee, Mr. T. W. Russell, somewhat suddenly announced a few days ago that the Committee considered they had received sufficient evidence, and that they proposed to close their inquiry and present their report. Very fortunately for the interests of the country, and very fortunately for everybody who held the position of Public Analyst in Great Britain and Ireland, Mr. Hehner had been heard, and he would strongly recommend every member of the Society who took an interest in the scientific and official work of Public Analysts to carefully read the evidence which Mr. Hehner had given on their behalf. It was the result, as he (Mr. Cassal) had reason to know, not only of very arduous and painstaking work, but of constant attendance, often at great personal inconvenience, at the meetings of the Food Products Committee, where one had to sit and listen to a good deal of what it would not be improper to call dreadful nonsense, to many ignorant or misleading statements, and, in fact, to much which could not but be extremely irritating to any member of that Society.

The questions that had to be dealt with were, as the Society knew, extremely complicated; and in view of the fact that some members of the Committee were decidedly hostile to the position which had been taken up by the Society of Public Analysts, Mr. Hehner's work was rendered all the more difficult. In the result, Mr. Hehner had proved himself a most worthy representative of the Society (hear, hear), and his evidence was admirable from beginning to end. There was one point touched upon which, as it was a matter of great importance, he might be pardoned for referring to. A question was put to Mr. Hehner as to what were the qualifications for admission to the Society of Public Analysts, and as to what steps, if any, were taken to ensure the restriction of membership of the Society to properly-qualified persons. It was hardly possible to explain to the Committee that the Society's system of proposal and election, properly applied—as it had been, at any rate, during recent years—was sufficient to ensure the exclusion of unqualified men, and that if, by any chance, there should be any such now in the Society, they must be very few. It was absurdly unjustifiable to draw the conclusion, which one member of the Committee appears to have drawn from Mr. Hehner's answers, that the Public Analysts as a body were not qualified to advise the Committee, or to put forward some of their number as fitted to sit on a Board of Reference and Control, because the Society's rules did not make it compulsory on candidates for election to have a scientific degree. He (Mr. Cassal) mentioned this matter because he felt that it was the duty of every member of the Society to see that no person was elected as a member of whom they would afterwards have reason to be ashamed; and he trusted that every member of the Society would be impressed with the full significance of

the question put to Mr. Hehner upon this matter, and with the great importance of always being able to answer such questions as Mr. Hehner had done, or in such a way that the position of the Society before the public should in no degree be weakened. He would just add—and he was sure that Mr. Hehner would bear out the statement—that as a Society they were also very greatly indebted to a member of the Committee who had done most excellent work during its sittings, and who would be found to have rendered a considerable public service by his attention to the matter. He referred to Mr. Hudson E. Kearley, M.P. He begged to move that the Society should thank Mr. Hehner for the great trouble that he had taken in preparing the evidence that he had placed before the Parliamentary Committee, and for the admirable way in which he had given that evidence on their behalf (applause).

Mr. ALLEN said he rose to second that motion with the very greatest pleasure. At some personal inconvenience, he had attended a number of the meetings of the Parliamentary Committee during the previous sessions. He did not hear Mr. Hehner give his evidence, but he had read every word of it on the proof. He thought the Society was indebted to Mr. Hehner in the highest degree for the masterly manner in which he had put their case before the Committee, and had met all the cross-questions and difficulties which constantly arose. They might congratulate themselves on having had their case laid before the Committee in the best possible way, and in a manner so complete as to leave very little else to be said. Of course, they regretted the premature termination of the Committee's labours. There were several points which not only Public Analysts, but others interested in the Food Acts, would have been glad to have brought before the Committee; but so far as the Society of Public Analysts were concerned, their case had been stated well, and they had no reason whatever to be dissatisfied with the impression made upon the Committee by Mr. Hehner's evidence. He had great pleasure in seconding the motion which Mr. Cassal had proposed.

The PRESIDENT said that, before putting the motion to the meeting, he would like to add his own word of grateful thanks to Mr. Hehner for the way in which he had attended the meetings of the Select Committee, and for the very able and admirable manner in which he had put the case of the Public Analysts before the Committee. He (the President) had read and re-read the evidence of Mr. Hehner, and the evidence of other analysts, and was quite sure that they had every reason to be very proud of Mr. Hehner, and he was sure that a deep impression had been made upon the Committee by him. The Council of the Society of Public Analysts had already passed a vote of thanks, and of their grateful appreciation of Mr. Hehner's services. He was sure that a similar vote would be passed at the present meeting with acclamation.

The resolution was carried unanimously, amid great applause.

Mr. HEHNER said that if he had known what was coming he would probably not have been present. He was very deeply grateful for the very kind manner in which the motion had been brought forward and carried. He did not require, however, any vote of thanks. He had identified himself with the Society for many years, and had spent so much thought and work upon it, that to break a lance for the Society had been a labour of love. He sincerely hoped that the views of the

Society, as represented by him, had made some impression upon the Committee. He desired to utilize this opportunity, instead of simply rising to thank the meeting, to give those members who were not at the meetings and had not had the recent reports of this Committee a summary of what passed, both as regards his own evidence and the reply to it by Mr. Bannister.

Mr. Hehner then gave a sketch of the evidence which he had recently given before the Select Committee of the House of Commons on Food Products Adulteration, and also of the evidence given by Mr. Bannister by way of rejoinder.

[A summary of both will be published in the ANALYST hereafter.—ED.]

THE BACTERIOLOGICAL EXAMINATION OF WATER FOR THE TYPHOID BACILLUS.

By T. H. PEARMAIN AND C. G. MOOR, M.A.

(Read at the Meeting, April 1, 1896.)

It is now universally acknowledged that polluted water is the most important agent in the conveyance of enteric fever. Although water contaminated with sewage has been, and is still, drunk by a large number of people with impunity, so far as the appearance of enteric fever is concerned, yet the slightest contamination of a water-supply with the dejecta from a case of typhoid fever, has, in many well-authenticated instances, caused widespread epidemics of the disease amongst those persons who had used the polluted water-supply.

In many of the recorded cases of water-borne typhoid, the amount of organic matter accompanying the specific pollution was so extremely small that the water-supplies have been repeatedly pronounced from the results of chemical analysis to be of high organic purity. Moreover, it has been shown that the organism which is the cause of enteric fever, may, when introduced into potable water of good quality, not only retain its vitality for a considerable period of time, but may multiply almost indefinitely. Therefore the slightest contamination with the alvine discharges from a case of true enteric fever may serve to render dangerous millions of gallons of drinking water. Thus it will be seen that the virulence of typhoid contaminated water is not necessarily dependent upon the organic impurity of the water, but upon the specific pollution. If this is granted, and experimental proof may be easily applied,* it will be admitted that under certain circumstances the question may arise, Has the epidemic of enteric fever now in progress in a given community had its origin in the water-supply? If this question be asked us, as analysts, we see that it is quite conceivable that the chemical analysis of the water by itself may be unable to afford evidence of pollution, owing to the accompanying organic matter being so

* A drop of a broth culture of *B. typhosus* (twenty-four hours old) was well diluted with sterile water. 1 c.c. of this diluted culture was added to 200 c.c. of the ordinary tap-water. The number of organisms was then estimated by an ordinary gelatin plate culture, when 1 c.c. of the water was found to contain approximately 900,000 organisms. This amount of pollution was not sufficient to raise the amount of albuminoid ammonia appreciably. The tap-water previously contained only 200 organisms per cubic centimetre.

trifling in quantity as to fall below the limits of experimental error. Are we then, to confess total inability to offer any assistance to discover and remedy the cause of the outbreak? Would not such a course of action as this simply lead to the calling in of others who profess to trust only to the bacteriological examination of water, and deny that the chemical examination has any value?

In short, what we are attempting to show is that every analyst who has to examine potable water should make himself thoroughly acquainted with the latest principles of bacteriological research bearing upon this particular point, so that he may be in a position to himself undertake the search for the specific organism in question with as much confidence in the results of his work as it is possible for one to feel in the present state of our knowledge.

It must be admitted that the proof of specific pollution in a number of the epidemics of water-borne typhoid rests on a somewhat incomplete basis, as will be seen from the perusal of an interesting series of papers by Ernest Hart, which have recently appeared in the *British Medical Journal*.* The bacillus of typhoid fever has, however, been isolated by many competent observers from water that had conveyed and caused the disease. Some doubt attaches to the identification of the organism by some of the earlier investigators, owing to the almost constant presence in the waters of other organisms so closely resembling the typhoid bacillus that their differentiation is a matter of great difficulty. The organism which has given rise to much confusion is the *B. coli communis*. This bacillus is a constant inhabitant of the intestinal tract, and the fæces of both man and animals, and therefore is almost invariably found in all polluted waters.

In order to ascertain whether the typhoid bacillus is present in any given water, care must be taken that the *B. coli communis* is not mistaken for the former. This is a very difficult matter, as the vitality of the bacillus coli communis is much greater under all conditions than that of the typhoid bacillus. The object is generally attained by the addition of various chemical substances to the nutrient media, which effectually inhibit or destroy the growth of organisms other than the colon and typhoid bacilli. As pointed out by Frankland, such additions have frequently destroyed the typhoid bacillus, and left the *B. coli communis*, owing to its greater power of resistance, alone master of the field.

According to many authorities, notably Messrs. Roux and Rodet, there is reason to believe that the *B. coli communis*, under certain conditions, such as growth in sewage, etc., assumes a pathogenic character, and gives rise to a disease which is clinically indistinguishable from enteric fever. This view is borne out to a great extent by the fact that water contaminated with faecal matter may be instrumental in causing typhoid fever without the actual access of the specific bacillus, as cases are on record where water long known to be polluted has acquired the property of conveying typhoid without the previous known contamination from a specific case of the disease.

This is in accordance with the well-established fact that in places where enteric

* *Water-borne Typhoid*.—A Historic Summary of the outbreaks in Great Britain and Ireland, 1858 to 1898. By Ernest Hart, *British Medical Journal*, June 15th, 22nd, 29th; July 6th, 13th, 20th; August 17th, 1895.

fever was once endemic, it has disappeared upon the substitution of a pure for a contaminated water supply.

Messrs. Demel and Orlandi* show that Roux and Rodet's statements as to the near relationship of the *B. typhosus* and the *B. coli communis* are borne out by the physiological and pathological properties of the metabolic products of the two organisms. Animals rendered immune to one are also rendered immune to the other bacillus, the virus of the *B. coli communis* having a higher therapeutic value than that of the *B. typhosus*.

Germano and Maurea,† after a very prolonged investigation, have isolated no less than thirty varieties of typhoid-like bacilli, and they consider this fact supports the theory that the *B. coli communis* may, under certain conditions, develop into the *B. typhosus*. Nicolle,‡ after a very careful investigation, could only find the *B. coli communis* in a typical case of enteric fever, the blood and spleen being particularly examined.

From the above facts it will be seen that the possible dangers to be derived from the drinking of sewage-polluted waters are enormously increased.

It is worth recording, however, in connection with the above, that Chantemesse has called attention to the fact that during the typhoid epidemic in Paris, in 1894, the soldiers who drank the polluted water supplied to the Ménilmontant barracks all escaped typhoid, notwithstanding the fact that the water was swarming with the colon bacillus.

Dr. Klein has recently studied§ the *B. typhosus* and *B. coli communis* as to their stability as separate species in culture, and in the process of transference from animal to animal. On the one hand bacilli of both kinds derived in each instance from human sources, were tested by him as to their vitality, and as to the retention of their differential characters in waters of different composition and quality. He also took the two organisms derived from sources outside the human body, namely from excrementally-polluted water supplies; these were passed from sub-culture to sub-culture, and were passed from peritoneum to peritoneum in separate series of guinea-pigs, they being cultured through no less than thirty generations. Whatever the source of the bacilli, and whatever the experimental conditions in the laboratory, or the animal body to which they were exposed, each organism retained unimpaired its differential characters, and at no time did they show the least tendency to depart from the characters generally accepted as being exhibited by these organisms. Incidentally, during the course of these experiments, it appeared that the persistence in a water medium of both the typhoid bacillus and the bacillus coli is largely governed by the chemical constitution of the water.

Dr. Elsner, of Berlin, has recently found the *B. typhosus* in the stools of a male nurse in perfect health who attended typhoid patients. The fact that a man in good health can carry the bacillus of enteric fever in his intestines, and thus

* *Centralb. für Bakteriologie*, xvi., p. 246.

† *Centralb. für Bakteriologie*, xv., p. 60.

‡ *Annales de l'Institut Pasteur*, 1895, No. 1.

§ *The 23rd Annual Report of the Local Government Board*—Supplement containing the Report of the Medical Officer, p. 459.

disseminate it, throws much light upon the so-called "spontaneous" origin of typhoid fever.

The bacillus of typhoid, or enteric, fever (*B. typhi abdominalis*) was first described by Eberth in the year 1883, who stained it in sections of the intestines of patients who had died of typhoid; in the following year, Gaffky obtained pure cultures of the organism, which is now known as the Eberth-Gaffky bacillus. The bacillus is 2.5 to 4.0 μ long by 0.5 μ thick.

The Eberth-Gaffky bacillus is not killed by drying, nor by exposure to a low temperature. Its thermal death-point is 55° C., and, according to most observers, it does not form spores. Like all the pathogenic organisms, it is prejudicially affected by light, diffused daylight being sufficient to prevent its development, while direct sunlight is fatal in five hours. The organism grows equally well both under aerobic and anaerobic conditions. The microscopical appearance alone is not enough to distinguish it from several other organisms—in fact, it is not uncommon to find some stained specimens which have a curved appearance exactly like the "Koch's Comma," or the Finkler-Prior bacillus. If a fragment of a recent culture is rubbed up with a droplet of water and examined with a twelfth-inch objective, the bacilli will be seen in active movement, this motility being due to the great number of hair-like flagella by which the organism is covered.

The typhoid bacillus stains well with all the usual basic aniline dyes, but is decolorized by Grams' method of staining.

The true Eberth-Gaffky bacillus is readily distinguished from all others by its characteristic growth on the various culture media. Repeated sub-culture, as in the case of many other organisms, produces longer and abnormal forms. Very lengthened forms of the bacillus, which are somewhat characteristic, are sometimes seen in cover-glass preparations; the long bacilli are known as "leptothrix" forms.

The *Bacillus coli communis* is slightly shorter than the typhoid bacillus, and, like it, owes its power of motion to flagella, but it never possesses the profusion of these usually seen in the case of the Eberth-Gaffky bacillus.

The following table shows the main points of difference between the typhoid bacillus and the colon bacillus for which it might be mistaken:

Media.	Bacillus typhosus.	Bacillus coli communis.
Gelatin plates ...	The colonies on the surface form large grayish-white expansions with irregular edges, and after a time become somewhat yellowish-brown. The depth colonies are darker, with regular edges. Under a low power the colonies exhibit a characteristic woven structure. The gelatin is not liquefied.	The colonies are round and oval, with smooth-rimmed margins. The surface-colonies form dirty white expansions, which, on magnification, exhibit a furrowed appearance. The colonies later become dirty yellowish-brown in colour. The gelatin is not liquefied.

Media.	<i>Bacillus typhosus.</i>	<i>Bacillus coli communis.</i>
Gelatin "streak" culture	Produces a grayish-white expansion with irregular edges. The growth has a tendency to keep to the inoculation streak, and often has a bluish iridescence.	Dirty yellowish white expansion, which spreads all over the surface of the media, and often has a bluish iridescence when viewed by transmitted light.
Gelatin "shake" culture	No gas bubbles.	Copious gas formation.
Agar-agar "streak" culture	Grayish - white expansion, which covers the surface of the medium.	Dirty yellowish-white expansion, which spreads over the surface of the medium.
Potatoes	Generally a faint grayish-white growth; the growth varies, however, on different potatoes.	Slimy yellowish growth.
Milk	Turns faintly acid. No coagulation takes place.	Curdled after one to three days.
Broth	Rendered turbid, and gives no indol reaction.	Rendered turbid, and gives a well-marked indol reaction after from twenty-four to forty-eight hours.
Formalin broth (1 : 7,000)	No growth.	Growth.

The growths make their appearance on the gelatin media at room temperature after from forty-eight to sixty hours. The agar-agar, potato, milk, and broth tubes are incubated at 37° C. (blood-heat) for three days.

From the above table it is seen that the *B. coli communis* is readily distinguished from the *B. typhosus* by its cultural characteristics, and particularly by its chemical reactions. The colon bacillus invariably grows more rapidly than the typhoid bacillus.

The colon bacillus when grown in alkaline peptone broth produces a red coloration on the addition of nitrous acid, depending upon the interaction of nitrous acid with indol, which is one of the products of the metabolism of the organism, to form nitro-indol, which is of a red colour. This test is best applied as follows: To 10 c.c. of the culture of the organism under examination, which has been incubated at blood-heat for not less than twenty-four hours, is added 1 c.c. of a 0.02 per cent. solution of sodium nitrite, and then a few drops of pure concentrated sulphuric acid. If indol is present, a rose to deep red coloration is produced.

The foregoing reaction, together with the power of the *B. coli communis* of coagulating milk and the formation of gas in nutrient gelatin, provide an easy method of distinguishing this organism from the *B. typhosus*.

When an organism which fails to coagulate milk is isolated from water by any of the methods hereafter given, and does not give rise to gas formation in a gelatin "shake" culture, and does not cause liquefaction of the gelatin in a

"streak" culture, and, moreover, yields a negative indol reaction, and, again, has the same morphological characters as the *B. typhosus*, it is at least indistinguishable from the Eberth-Gaffky bacillus, and may with every probability be regarded as identical with it. Animals do not suffer from enteric fever in the manner in which we know it in man, hence inoculation experiments do not give us any assistance in proving a given organism to be identical with the specific bacillus of typhoid fever. Dr. Klein* has recently pointed out that, although the Eberth-Gaffky bacillus is regarded as the essential cause of enteric fever, it has never yet been experimentally proved capable of inducing that disease. Rodents inoculated, whether subcutaneously or intra-peritoneally, with this bacillus do indeed suffer fatal illness; but the malady thus induced is not of a specific nature, and, moreover, may just as easily be produced by the injection of various other microbes. He has shown that the Eberth-Gaffky bacillus may grow and multiply within the inguinal lymph glands of calves.

A number of organisms have been described by Cassedebat, Babes, Booker, Klein, Springthorpe, and others, which were obtained from cases which were clinically identical with enteric fever and other sources which resembled the Eberth-Gaffky bacillus, but were shown to present slight but constant differences in their cultural characters. They are only to be differentiated from the true typhoid bacillus by a very careful comparison of cultures made side by side on various media. Cassedebat found three species of these so-called "pseudo-typhoid" bacilli in the Marseilles water-supply during the great typhoid epidemic in that town during 1891. They all corresponded with the Eberth-Gaffky bacillus in their growth upon gelatin, potato, etc., and they all gave a negative indol reaction. Like the typhoid bacillus, they grew in milk without causing the coagulation of the caseine, but two of them produced an alkaline reaction in this medium, while the third corresponded with the Eberth-Gaffky bacillus in producing a decided acid reaction.

It does not seem impossible that these pseudo-typhoid organisms may be the colon bacillus in transition stages.

(To be continued.)

THE ESTIMATION OF THE DIASTATIC POWER OF MALT, ETC.

By W. J. SYKES, M.D., AND C. A. MITCHELL, B.A.

(Read at the Meeting, March 4, 1896.)

AMONGST the different items which have to be ascertained in the examination of malt, the determination of its diastatic power is one of considerable importance, since the requirements of manufacturers in this direction vary considerably, according to the nature of the article they wish to produce.

Regarded from a diastatic point of view, malts may roughly be divided into four classes, in a gradually descending scale:

Green Malt.—This is barley fully germinated, and ready for placing on the drying-kiln. Since malt invariably suffers a loss of diastatic power during the drying process, however carefully and in whatever way this may be conducted, some

* See the 22nd and 23rd Annual Reports of the Local Government Board—Supplement containing the Report of the Medical Officer.

distillers, in order to avoid this loss, make use of malt in this form. By doing so they not only avail themselves of malt possessing the highest possible diastatic power, but at the same time save the cost of drying.'

Air-dried Malt.—This is green malt dried at the ordinary temperature in a current of air. It is mostly used for experimental purposes, such as the preparation of diastase, etc.

Distillers' Malt.—In this the germination of the barley is pushed so far as to develop the diastatic power of the grain to its maximum degree. The green malt so produced is then dried slowly and carefully on the kiln, where it is never allowed to reach a temperature higher than 120° to 125° F. In this way loss of diastatic power is reduced to the minimum consistent with the production of a staple article.

Brewers' Malt.—In this the germination is not carried so far as in the before-mentioned malts, consequently the diastatic power is not developed to the fullest extent. Though in the initial stages of the drying process the heat is applied very slowly and gradually, towards its termination the temperature is allowed to rise, for pale malts, to 180° F., and for high-dried malts to as much as 210° to 220° F. The malt in these latter stages undergoes a sort of cooking process, which largely develops its flavour and aroma. During this portion of the drying much of its diastatic power is lost, but with malts intended for the brewing of beer, it is necessary that such should be the case.

From time to time various processes have been proposed for the estimation of the diastatic power of malt, many of which are of a more or less crude and unsatisfactory nature. The first really efficient process was that devised by Kjeldahl (*Comptes rendus des Travaux de Carlsberg Lab.*, 1879). In a series of investigations on the action of diastase on starch, he had discovered that in such transformations the amount of reducing substance formed was strictly proportional to the amount of diastase, so long as the amount produced in the mixture was not allowed to exceed 45 per cent. of the total possible yield. In other words, to comply with what is now known as "Kjeldahl's law of proportionality," the diastase must not be present in a quantity greater than will convert a little less than half the starch present.

For the actual determination, 10 grammes of the malt, after being finely ground, were extracted with 400 c.c. of water for six hours; 15 c.c. of the bright filtrate from this were added to 200° c.c. of a 3 to 4 per cent. solution of soluble starch, heated to a temperature of from 57° to 59° C., at which temperature the whole was kept for twenty minutes. The amount of reducing substance reckoned as maltose was then determined either gravimetrically, volumetrically, or colorimetrically, and from this the amount of reducing substance contained in the original soluble starch solution was deducted. The diastatic power was considered to equal 100, when 15 c.c. of malt-extract of the above strength and under the above conditions yielded an increase of cuprous oxide equivalent to 1 gramme of maltose. The soluble starch used in Kjeldahl's process was prepared by allowing an aqueous extract of malt to act upon gelatinized starch at a high temperature (180° F.). In such a case, those portions of the diastase, which convert the starch into bodies capable of reducing Fehling's solution, are almost completely destroyed, and only the liquefying portion of the diastase remains intact—that is, the portion which converts gelatinized starch into its soluble

modification. However carefully the operation may be performed, some reducing bodies are invariably produced, hence it is necessary to determine their amount in each fresh batch of solution employed; and as soluble starch solution is unstable, it must be prepared as wanted, and this is a distinct inconvenience.

Some ten years later C. J. Lintner discovered that soluble starch could be readily prepared in a stable form by the limited action of various acids upon starch, a fact which he practically applied to the simplification of Kjeldahl's process.

Lintner's soluble starch is prepared by allowing potato starch to stand under 7.5 per cent. hydrochloric acid for seven days at the ordinary temperature, or for three days at a temperature of 104° F. The same result can be obtained, according to Brown and Morris (*Trans. Chem. Soc.*, 1889, p. 450), in twenty-four hours, at the ordinary temperature, by employing 12 per cent. hydrochloric acid. The starch is then washed until free from acid, dried in the air, and preserved for use. Under this treatment the starch completely loses its power of forming a paste; and though it is almost insoluble in cold water, is readily soluble in that medium at temperatures over 60° or 70° C., from which it again very slowly separates on cooling. On microscopic examination no change can be detected in the appearance of the starch granules either with ordinary or with polarized light. Its solution has not the slightest reducing action upon Fehling's solution.

For the estimation of the diastatic power of a substance, a 2 per cent. solution of this soluble starch is employed. It is best prepared by placing the required amount of water in a flask, and the weighed quantity of soluble starch in a smaller beaker. Sufficient water is poured from the flask on to the soluble starch to form a mixture of the consistence of thick cream, the remainder of the water in the flask brought to near its boiling point, and the well-stirred contents of the beaker poured in, when the soluble starch dissolves immediately. A little of the hot solution is poured into the beaker to dissolve any remnants of starch, and returned to the contents of the flask, which are then allowed to cool.

Twenty-five grammes of malt are finely ground in a coffee-mill and stirred into 500 c.c. of water. The mixture is allowed to stand for six hours, with an occasional stir. The solution is filtered through a dry filter-paper, the first cloudy portions rejected, and about 50 c.c. of the bright filtrate (malt-extract) collected.

Ten test-tubes, of a capacity of about 20 c.c., are taken, and 10 c.c. of the cold 2 per cent. soluble starch solution pipetted into each. To the first of these is added 0.1 c.c. of the malt-extract, to the second 0.2 c.c., and so on, so that the tenth test-tube receives 1.0 c.c. After each tube has been well shaken, the whole are allowed to stand for one hour in a water-bath kept at a temperature of 70° F. At the expiration of this period, 5 c.c. of Fehling's solution are added to each, the tubes well shaken, and immersed in boiling water for twenty minutes, after which they are taken out and examined. It will perhaps be found that the contents of one of the tubes are colorless, in which case the amount of malt-extract added to this tube is taken for the subsequent calculation. Oftener the contents of one tube are yellow, the next one blue; in this case the average of the amounts added to the two tubes is taken.

The diastatic power is calculated on the basis that when 0.1 c.c. of malt-extract

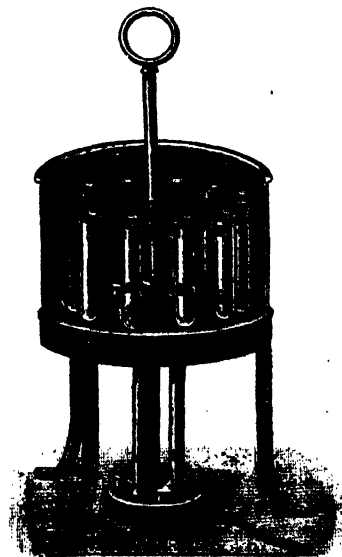
of the above strength added to 10 c.c. of 2 per cent. soluble starch solution, and treated in the above manner, yields just sufficient reducing substance to exactly reduce 5 c.c. of Fehling's solution, the diastatic power of the malt yielding the extract shall be taken as 100. The diastatic power of any other malt is found by a sum in inverse proportion. For instance, if in such a determination the end point is found to lie between the sixth and seventh tube, the amount of malt-extract to be taken would be $0.6 + 0.7 \div 2 = 0.65$, and $\frac{100}{0.65} \times 0.1 = 15.55$. This figure must then be calculated upon water-free malt: supposing the malt contained 4.5 per cent. of moisture, then $\frac{100}{95.5} \times 15.55 = 16.23$ expresses the diastatic power of the malt on Lintner's scale.

In the examination of green malts, where the diastatic power often exceeds 100, the malt-extract is diluted with an equal volume of water before being added to the soluble starch tubes. The result is then multiplied by 2.

For determining the diastatic power of precipitated diastase, a solution of 0.03 grammes of the substance in 25 c.c. of water takes the place of the malt extract. Consequently, the energy of a precipitated diastase is considered as 100 when 0.12 milligramme yields just sufficient reducing substance to decolorize 5 c.c. of Fehling's solution under the above experimental conditions. This quantity was fixed upon because it represented the power of the most energetic diastase which had been then obtained. In recent times Osborne (ANALYST, xx. 232) has prepared a diastase six hundred times as energetic as this. In examining such specimens, the diastase solution has to be made correspondingly weaker; the right proportion is found by preparatory trials of solutions of various strengths, in which 0.1 and 1.0 of the solution are respectively added to 10 c.c. soluble starch solution. When one of these after treatment with Fehling's solution shows over, and the other under, reduction, the solution of diastase is of the proper strength. The degree of dilution must obviously be taken into account in the subsequent calculation.

For conducting experiments of this kind, the star-apparatus of Reischauer, illustrated in the accompanying engraving, is exceedingly convenient. A special pipette, graduated so as to deliver quantities gradually increasing from 0.1 c.c. to 1.0 c.c. by 0.1 c.c., is also useful.

It is obvious that in the above method there are only ten definitely fixed points, and consequently a definite result can only be obtained in the operation when one of the tubes is exactly decolorized, and this rarely happens in practice. Should the end point lie between two tubes, in order to obtain a more approximate result the operation must be repeated with gradually increasing quantities of malt-extract, commencing with the quantity which caused incomplete reduction,



and ending with that which produced over-reduction. Should the end-point again fall between two tubes, the operation has to be performed a third time, and so on.

In the determination of the diastatic power of brewers' malts, which range from 25 to 45, since the end-point does not fall between the first and second tube, the numbers found are, as a rule, sufficiently approximate for practical purposes, and in any case do not require more than one repetition of the operation. With distillers' malts the case is different; the end-point generally falls between the first and second tube, and between these there is a wide gap—from 50 to 100.

In order to avoid several repetitions of the operation, and to obtain the desired result in one determination, we suggest a slight modification, which to some extent combines the methods of Kjeldahl and Lintner. The soluble starch solution, malt-extract, and diastase solution, are all prepared in the manner mentioned before. Instead, however, of employing a number of tubes, we make use of one small wide-mouthed flask, of a capacity of about 200 c.c. Into this 100 c.c. of the soluble starch solution are placed, 1.0 c.c. of the malt-extract added, the whole well shaken, and allowed to stand at a temperature of 70° F. for one hour; 50 c.c. of Fehling's solution are then added, the flask, with its mouth covered with a watch-glass,* placed on wire-gauze over a Bunsen-flame, and heated up to 98° C. It is then removed from the flame, and immersed in boiling water for seven minutes. It has been found experimentally that by proceeding in this way the contents of the flask are submitted to conditions of temperature and time closely approximating those of the test-tubes employed in Lintner's method. The cuprous oxide is collected in a Soxhlet filter-tube, reduced in hydrogen, and weighed. The weight of the copper found, divided by 0.438 (the quantity of copper contained in 50 c.c. of Fehling's solution), and multiplied by 100, gives the diastatic power. In this way a determinate figure is obtained for any diastatic power between 0 and 100 by one operation.

In the following table we give the results of a number of experiments which were made with a view to determining how the figures obtained in this way agree with those obtained by Lintner's method:

Malt.	Moisture per cent.	Extract lbs. per quarter.	Diastatic Power.		Remarks.
			Lintner's Method.	Sykes and Mitchell.	
1. English ...	—	—	5	3.3	Old musty sample.
2. Smyrna (Black and White, 1892)	—	—	10	11.2	
3. Foreign ...	3.8	85.34	12	15	
4. Syrian ...	6.7	78.96	12	15.7	
5. English ...	12.7	—	17	22	Old sample.
6. Syrian ...	4.01	85.0	19	22.7	
7. English ...	4.17	90.72	21	22.8	Said to be low dried.
8. Yorkshire (1892)	10.1	—	25	24	
9. Foreign ...	7.3	77	40	38.7	
10. English ...	4.7	94	77.2	83	Dried at 125° to 130° Fahr.

* Bearing in mind Kjeldahl's experiments (ANALYST, xx. 227), in some cases a current of hydrogen was passed through the flask during the heating process, in others the flask was simply covered with a watch-glass. This latter precaution was found sufficient to prevent the disturbing influence of the atmospheric oxygen.

The time required for making an estimation, amounting to about eight hours in all, is often a very inconvenient one; but we have found that the malt-extract may be filtered after the six hours, and the filtrate left until the next day without affecting the result. This is shown by the results given in the following table:

Sample.	Estimation immediately after filtering.			Estimation next day.		
		Lintner's.	S. and M.		Lintner's.	S. and M.
3	...	12	15	...	14	16
5	...	17	22	...	16	18
6	...	19	22.7	...	19	—

From these examples it will be seen that the figures obtained by our method agree fairly well with those obtained by Lintner's. It is probable that the former are the more accurate, since under the conditions of the process Kjeldahl's law of proportionality is strictly adhered to, and the figures obtained by Lintner's method are often the average between two tubes.

As the whole process is to a great extent of an empirical nature, the results obtained by either modification would possess the same utility for practical purposes. We can, however, fairly claim that a closer result is obtained in a shorter time by our method, and that it is especially useful when malts having high diastatic powers have to be dealt with.

DISCUSSION.

Mr. A. R. LING said that he was glad to hear that the authors regarded the method as a purely empirical one, and, in his opinion, it must remain so. It had been stated by several observers—and his own experiments in conjunction with Mr. J. L. Baker bore out that statement—that when some malts were made to act on starch paste, a certain amount of glucose was found among the products of the transformation, and, in the case of these malts, of course, Kjeldahl's law of proportionality—on which the method of determining diastatic powers was based—would not hold. In the method as usually carried out the diastatic solution (malt extract) was allowed to act on the solution of soluble starch at practically the ordinary temperature. But, inasmuch as the mashing temperature adopted in brewing was considerably above this, he thought that the determination of the diastatic capacity of a malt at the particular temperature at which it was to be afterwards mashed would perhaps furnish information of greater practical utility. He had used an ordinary titration method for determining the diastatic power of malt, and found that it yielded results which compared favourably with those obtained by Lintner's method. With regard to malts of very high diastatic power, in Lintner's method the value indicated between the first two tubes—containing 0.1 and 0.2 c.c. of malt extract respectively—was 100–50; and he should like to know the accuracy which could be attained by the author's method with malts of this class.

Mr. A. C. CHAPMAN asked if the authors could give any information as to the working of the process in connection with distillers' malts of very high diastatic power, viz., higher than 77, which was the highest mentioned in the table, as he understood that the process had been devised more particularly to meet the requirements of such cases. In the examples given the agreement of the numbers with those obtained by Lintner's method was certainly good. He also pointed out that if it were permissible to allow the aqueous malt extract to stand overnight in this case, the same might be

said of Lintner's method, for the experiments were up to this point carried out in practically the same manner in the two methods.

The suggested correction for the reducing substance in the aqueous malt extract had always appeared to him to be unnecessary in view of the limits within which it was possible to reach the differences between the various tubes.

With regard to the estimation of the amount of copper obtained, he had obtained perfectly satisfactory results by weighing the cuprous oxide direct, which was of course much simpler and quicker than reduction in hydrogen. He used a platinum Gooch's crucible, and filtered off and washed the oxide with the aid of a filter pump. The time occupied in filtering, washing, drying and weighing, did not amount to many minutes.

Dr. SYKES, in reply, said that although the method they had described, as with all others on the same principle, could not be said to be an absolute measure of the diastase, still, since Lintner's figure was usually an estimate between two tubes, he thought the figures they obtained were more determinate.

With regard to the reducing bodies formed, he thought it was exceedingly probable that they did vary materially in different cases, but, as the whole subject of the transformation of starch under the action of diastase was still involved in considerable obscurity, it was impossible to say what the differences were.

Mr. Ling's proposal to conduct the diastase determinations at the temperature at which the malt would be used in actual practice, was certainly a point worthy of consideration. It might probably be found that such a course would yield results more in accordance with the behaviour of the malt in the mash-tun.

With regard to Mr. Chapman's inquiry about distillers' malts, such samples seemed somewhat difficult to meet with, and the figures referred to in the tables were the highest they had been so far able to obtain.

There was no objection to letting the filtered malt extract remain overnight when Lintner's process was employed.

They had estimated the copper by reduction in hydrogen, because the almost universal consensus of opinion appeared to favour the view that this was the most exact method. If no oxidation of the cuprous oxide took place in Mr. Chapman's method, then, by an alteration of the factor, it would serve equally well.

OFFICIAL METHODS FOR THE ANALYSIS OF FERTILIZERS, ISSUED
BY THE GERMAN MANURE MANUFACTURERS' ASSOCIATION,
HARZBURG, MAY 28, 1895.

CONTRIBUTED BY H. H. B. SHEPHERD, F.I.C., ANGLO-CONTINENTAL GUANO WORKS,
LONDON.

(Continued from p. 101.)

3. *The Molybdate Method.*—*Preparation of the Molybdenum Solution.**—One hundred grammes of pure molybdic acid are dissolved in 400 grammes of a 10 per cent. solution of ammonia (0.960 specific gravity), and this solution is mixed while

* According to the Berlin resolutions of December 14, 1892; Halle, 1893.

shaken with 1,500 grammes nitric acid of 1.2 specific gravity. It is then placed in a water-bath heated to 50° C. for one hour, and afterwards left for two or three days in a warm place, to ensure the precipitation of any phosphoric acid that may be present. It is desirable not to allow the temperature to exceed 50° C., as at 90° C. the separation of molybdic acid is very considerable.

Preparation of the Magnesia Mixture (the Halle formula, containing an increased quantity of ammonia).—Five hundred and fifty grammes magnesium chloride, and 1,050 grammes ammonium chloride, are dissolved in water with 3½ litres of concentrated ammonia (24 per cent. = 0.91 specific gravity), and the mixture made up to 10 litres.

Determination.—For materials (superphosphates or raw-phosphates) containing up to 20 per cent. phosphoric acid, 50 c.c. of the solution = 1 gramme substance, are to be taken; and for materials above 20 per cent. phosphoric acid, 25 c.c. = ½ gramme substance. Two hundred c.c. molybdenum solution are added, and the solution digested on the water-bath for three hours at 50° C. It is then cooled, and the yellow precipitate filtered off through a small funnel, care being taken not to allow more of the precipitate than possible to pass on to the filter. The precipitate is then repeatedly washed by decantation with a mixture of 100 parts of the above-mentioned molybdenum solution, 20 parts nitric acid of 1.2 specific gravity, and 80 parts water, until lime is no longer found in the washings. Five washings with 20 c.c. appear to be sufficient. The test for lime is the appearance or not of turbidity on adding a little alcohol acidified with sulphuric acid to 1 c.c. of the washings. The funnel containing the yellow precipitate is then placed over the vessel in which the precipitation took place, and the filter washed with the smallest possible quantity of a warm 10 per cent. ammonia solution until the precipitate completely dissolves. By careful working, this can be accomplished in three or four washings. The filter is then washed sufficiently (seven or eight times) with hot water. If the ammonia used in washing is insufficient to dissolve the yellow precipitate in the beaker, more must be added, and should the solution not be clear, it must be passed a second time through the same filter. The solution (which will be still warm) is as nearly as possible neutralized with hydrochloric acid (until the precipitate produced on the addition of a drop redissolves with difficulty), cooled, 25 c.c. of a 5 per cent. solution of ammonia added, and 20 c.c. magnesia mixture added drop by drop. After two hours' standing (or half an hour's shaking with the shaking machine), the precipitate is filtered off and washed with a 5 per cent. solution of ammonia until chlorine is no longer found in the filtrate. The precipitate is dried at 100° C., transferred to a platinum crucible, the filter-paper separately burnt, and the whole incinerated over the flame of a powerful burner. By the use of the Gooch (perforated) crucibles, the precipitate is both dried and ignited in the crucible. The crucible is finally heated for five minutes in a muffle or over the blow-pipe, taking care that if the blow-pipe is used, reducing gases do not find their way into the crucible. It is then cooled and weighed. The ignition must be repeated until the weight remains constant.

4. *The Citrate Method.*—*Preparation of the Solution.*—Five hundred and fifty grammes of pure citric acid are dissolved in 2,000 grammes of a 24 per cent.

ammonia solution (0.91° specific gravity), made up to 5 litres with water and filtered.

Determination.—For substances containing up to 20 per cent. phosphoric acid, 50 c.c. of the prepared solution, corresponding to 1 gramme of substance, are to be taken; for substances containing more than 20 per cent. phosphoric acid, 25 c.c. = 0.5 gramme of substance. Fifty c.c. citrate solution are added, and 25 c.c. magnesia mixture (as described under the molybdate method). The whole is then either stirred in a beaker, or shaken in an Erlenmeyer flask, for half an hour, allowed to stand for one hour and filtered. The 25 c.c. magnesia mixture should be added drop by drop, and allowed to fall into the middle of the liquid, to counteract the tendency of the precipitate to attach itself to the sides of the vessel. The ammonium magnesium phosphate is collected on a filter, as directed for the molybdate method, and washed with a 5 per cent. ammonia solution until the washings are free from lime and chlorine. The precipitate is further treated—i.e., dried, ignited, and weighed—exactly as described for the molybdate method.

5. *The Uranium Method.*—This method, though superseded, is nevertheless useful for testing superphosphates if moderately free from iron and alumina. By careful working, reliable results can be very quickly obtained with it, and on this account it is recommended for factory purposes.

Fifty c.c. of the solution, corresponding to 1 gramme of substance, are placed in a beaker and 12.5 c.c. of an acid solution of ammonium acetate added. When the precipitate produced has subsided, the liquid is filtered into a 400 c.c. or 500 c.c. flask and the precipitate washed with hot water until the washings are neutral. Standard solution of uranium nitrate is then added to the filtrate and washings, and the whole raised to the boil. The addition of the uranium solution is now continued until a drop or two taken out gives the usual reaction with potassium ferrocyanide. The quantity of uranium solution used indicates the percentage of phosphoric acid. The filter containing the precipitate is then burnt in the usual way and weighed, half its weight being taken as phosphoric acid and added to the phosphoric acid found by titration.

Preparation of the Reagents. — Uranium Solution.—Five hundred grammes chemically pure uranium oxide are dissolved in 13 to 14 litres of water to which 50 c.c. of concentrated nitric acid (1.4 specific gravity) have been added. The value of this solution is found by standardizing with a solution of monocalcium phosphate in which the phosphoric acid has been determined gravimetrically in 50 c.c. A superphosphate solution free from iron may also be used for this purpose.

The solution of monocalcium phosphate can be prepared by digesting 5.5 grammes of dry tricalcium phosphate in dilute sulphuric acid, making up to a litre with water and filtering off the gypsum and insoluble matter.

Acid Solution of Ammonium Acetate.—One hundred grammes of chemically pure ammonium acetate are dissolved in 900 c.c. of water and made up to a litre with glacial acetic acid.

Solution of Potassium Ferrocyanide.—This solution, which must be freshly prepared every time before use, is made by dissolving $\frac{1}{2}$ gramme chemically pure

potassium ferrocyanide in about 40 c.c. of water. A good light is required for discerning the colour.

6. *Determination of Citrate-soluble Phosphoric Acid.—Wagner's Method.*—Five grammes of superphosphate or precipitated phosphate are rubbed into a paste with diluted citrate solution (prepared as directed) and washed into a half-litre flask. It is then made up to the mark with the diluted citrate solution, allowed to stand for about eighteen hours at the ordinary temperature (17° to 18° C.), with frequent shaking, and filtered. (Thirty minutes in a shaking or stirring machine may, however, be substituted for the eighteen hours' standing.) Fifty c.c. of the filtrate are then taken, and molybdenum solution added in the proportion of 1 c.c. molybdenum solution for every milligramme of phosphoric acid, and to this is added one-fourth of its volume of concentrated ammonium nitrate solution (to be presently described). It is then placed in a water-bath heated to 50° C. for about twenty minutes, taken out, cooled, and the precipitate filtered off and washed with dilute solution of ammonium nitrate. A hole is then made in the apex of the filter, and the precipitate washed back into the beaker used for the precipitation by means of a $2\frac{1}{2}$ per cent. solution of ammonia. The filter is well washed, and 20 c.c. magnesia mixture added to the ammoniacal solution drop by drop, with constant stirring. After standing for about an hour, the precipitate is filtered off, washed with a 2 per cent. ammonia solution, and dried and ignited in the usual way.

Preparation of the necessary Solutions.—(1) *Concentrated Citrate Solution.*—One hundred and fifty grammes of citric acid are dissolved in water in a litre flask and the solution neutralized with ammonia. Ten grammes of citric acid are then added, and the whole made up to the mark with water.

(2) *Dilute Citrate Solution.*—One volume of concentrated citrate solution diluted with four volumes of water.

(3) *Concentrated Ammonium Nitrate Solution.*—Seven hundred and fifty grammes of ammonium nitrate are dissolved in water, and the solution made up to a litre.

(4) *Molybdenum Solution.*—One hundred and fifty grammes of ammonium molybdate are dissolved in water, the solution made up to a litre, and mixed with a litre of nitric acid of 1.2 specific gravity.

(5) *Magnesia Mixture.*—One hundred and ten grammes of pure crystallized magnesium chloride and 140 grammes of ammonium chloride are dissolved in 1300 c.c. of water, and 700 c.c. (8 per cent.) ammonia added.

*Belgian Method (Petermann's).**—*Quantities to be Taken.*—For superphosphates containing more than 20 per cent. phosphoric acid and for precipitated phosphates, 1 gramme; for superphosphates with 10 to 20 per cent. phosphoric acid, 2 grammes; for superphosphates with less than 10 per cent. phosphoric acid, and for compound manures, 4 grammes.

Extraction.—The weighed portion is first rubbed up in a glass mortar. It is then very thoroughly rubbed up with a small quantity of water and filtered by decantation into a 250 c.c. flask. After being treated three times in this way with water, it is afterwards washed on to the filter, and there further washed until filtrate

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and washings together amount to about 200 c.c. The filtrate and washings are then made up to the mark with water.

The filter with the insoluble residue is placed in a 250 c.c. flask, and 100 c.c. of Petermann's alkaline ammonium citrate solution added.

The digestion of the residue in the citrate solution is carried out in two stages. First, it is allowed to stand for fifteen hours at the ordinary temperature, with occasional shaking, and afterwards digested for one hour at 40° C., the time being reckoned from the moment when the thermometer in the water-bath registers this temperature.

Precipitation.—The solution is cooled, filtered, and made up to 250 c.c., and 50 c.c. of this mixed with 50 c.c. of the above aqueous solution. (If it has become turbid, a few drops of nitric acid should be added.) The combined water-soluble and citrate-soluble phosphoric acid is then determined in the mixture, either by the molybdate or by the citrate method.

Preparation of the Petermann Solution.—Five hundred grammes of citric acid are exactly neutralized with ammonia of 0.92 specific gravity; about 700 c.c. are required. This is then cooled, adjusted to 1.09 specific gravity at 15° C., 50 c.c. of ammonia of 0.92 specific gravity added for each litre, and the whole filtered.

7. *Determination of Phosphoric Acid and Percentage of Fine Meal in Thomas Phosphate.*—A. *Determination of Total Phosphoric Acid.*—(a) *Hydrochloric Acid Method.*—The sample is prepared for analysis by being rubbed up in a mortar and sifted through a No. 60 sieve (German gauge). Should any considerable quantity of iron remain, it must be weighed and taken into account. Ten grammes of the prepared sample and about 80 c.c. of concentrated hydrochloric acid are placed in a 50 c.c. flask and evaporated on a sand-bath until the solution is of about the consistence of syrup. A few drops of hydrochloric acid are then added, the solution cooled, made up to the mark with water, and filtered. Fifty c.c. of the filtrate are mixed with 100 c.c. of Maercker's ammoniacal citrate solution (1,500 grammes citric acid and 5,000 c.c. 24 per cent. NH_3 made up to 15 litres) and 25 c.c. magnesia mixture, and placed under the stirring machine for half an hour. It is then allowed to stand for two hours, filtered, and treated as described for the determination of the water-soluble phosphoric acid.

(b) *Sulphuric Acid Method.*—Ten grammes of the phosphate are first thoroughly mixed with a few c.c. of dilute sulphuric acid (1 in 2), and then 50 c.c. of concentrated sulphuric acid are added. The mixture is then raised to the boil, and kept at a heat just below its boiling-point until evaporated to a thick fluid, which commences to bump violently. It is then made up with water, and further dealt with by the molybdate or the citrate method.

(To be continued.)

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD ANALYSIS.

The Estimation of Alcohol and Extract in Wine by the Refractometer.
E. Riegler. (*Zeit. anal. Chem.*, 1896, 27-31.)—In this method the refractive power of the wine, of the residue freed from alcohol and made up to the original volume, and of distilled water are determined by means of a Pulfrich's refractometer, and from these data the results are calculated.

The refractive power of the wine = N consists of three parts :

1. That corresponding to the distilled water = a .
2. „ „ „ extract = b .
3. „ „ „ alcohol = c .

From these :

and $N - (a + b) = C$ = the part corresponding to the alcohol,
 $(a + b) - a = b$ „ „ „ extract.

From the examination of a large number of samples the author has obtained the following factors which he uses in the calculation :

1. One gramme of extract in 100 c.c. of wine raises the refractive power by 0.00145 as compared with that of water.

2. One gramme of alcohol in 100 c.c. of wine raises it by 0.00068.

In making the determination 25 c.c. of the wine are evaporated to about 8 c.c., and the residue made up again to 25 c.c. The flasks containing this extract solution, the original wine, and the distilled water are placed in a vessel of water at the temperature of the room, so that all three liquids have the same temperature.

The difference between the refractive power of the wine, N , and that of the extract solution $(a + b)$, divided by 0.00068, gives the amount of alcohol in grammes in 100 c.c. of wine :

$$\frac{N - (a + b)}{0.00068} = C.$$

The difference between the refractive power of the extract solution $(a + b)$ and that of the water, a , divided by 0.00145, gives the amount of extract in grammes in 100 c.c. of wine :

$$\frac{a + b - a}{0.00145} = \frac{b}{0.00145}.$$

Example :

Refractive power of wine	= 1.34105
Refractive power of extract solution	= 1.33550
Difference	= 555
555	
68	= 8.1617 grammes alcohol in 100 c.c. wine.
Refractive power of extract solution	= 1.33550
„ „ distilled water	= 1.33263
	287
287	
145	= 1.9793 grammes extract in 100 c.c. wine.

The following table gives some of the results obtained in this way, compared with those obtained by the ordinary methods :

Sp. Gr. at 15° C.	Extract in 100 c.c. Gra.	Alcohol in 100 c.c. Gra.	Refractive Power.			Refraction raised as compared with Water by 1 Gr. Ex- tract in 100 c.c. Wine.	Refraction raised as compared with Water by 1 Gr. Alcohol in 100 c.c. Wine.	Extract Calcu- lated. Gra. per 100 c.c.	Alcohol Calcu- lated. Gra. per 100 c.c.
			Wine = N.	Extract Solution = (a + b).	Of Dis- tilled Water = (a).				
0.9963	2.0660	7.0206	1.34037	1.33558	1.33254	0.00147	0.00068	2.0965	7.0441
0.9921	1.8800	9.4793	1.34165	1.33517	1.33237	0.00148	0.00068	1.9310	9.5294
0.9932	1.6240	7.7400	1.33978	1.33459	1.33238	0.00137	0.00067	1.5242	7.6324
0.9930	1.8784	8.6443	1.34088	1.33500	1.33222	0.00148	0.00067	1.9172	8.6470

The author considers this method equally suitable for the examination of beer, but has not been able to test the results on a sufficient number of samples to enable him to speak positively.

C. A. M.

The Estimation of Lævulose in Honey or Other Substances. H. W. Wiley. (*Jour. Amer. Chem. Soc.*, 1896, 81-91.)—The principle of the process depends on the change in the specific rotatory power of lævulose at different temperatures. In the author's apparatus the observation-tube was so constructed that it could be kept at the selected temperatures (zero and 88° C.) by means of a control medium, either water heated by a steam-jet or by ice. To avoid the deposition of moisture on the cover-glasses at the lower temperature, a tube of hard indiarubber was attached by means of a threaded screw to the ends of the observation-tube. This had a central axis of perforated brass of the same dimensions as the diameter of the observation-tube, outside of which were placed fragments of calcium chloride. The end of the tube was covered air-tight with an ordinary cover-glass. At the higher temperature an even temperature was maintained by means of a mechanical stirrer.

Before making a polarimetric reading the observation-tube should be kept for at least half an hour (sometimes longer), noticeably in the case of honey, at the required temperature. When solutions are to be read at a high temperature, the water surrounding the tube should have been recently boiled, so as to avoid the evolution of air-bubbles. A correction is made for the linear expansion and contraction of the tube at the temperature of observation.

The general formula for the calculation of the percentage of lævulose is :

$$L = \frac{R}{K(T-t)} \div W.$$

Where

K = deviation in divisions of cane-sugar scale or in angular rotation produced by 1 gramme of lævulose for 1° temperature.

T and t = temperatures of observation.

R = observed deviation in rotation.

W = weight of lævulose obtained.

L = percentage of lævulose required.

For most genuine honeys the value of R between 0° and 88° C. is approximately thirty divisions of the cane-sugar scale or 10° angular measure for 26.048 grammes in

100 c.c. read in a 200 mm. tube, or for 13.024 grammes in 100 c.c. read in a 400 mm. tube.

Example.—26.048 grammes of honey taken. The angular variation produced by 1 gramme of levulose for 1° of temperature is 0.01256°. For 88° C. this would be 1.10528°. In the given case the angular deviation observed between 0° and 88° C. was 10.404°. The weight of levulose present was $10.404 \div 1.10529 = 9.413$ grammes, and the percentage = 16.13.

In cooling solutions of honey the maximum left-handed rotation is not reached as soon as the temperature reaches zero, but only after it has been kept at that temperature for two or three hours. They should therefore be left for that time before taking the reading.

C. A. M.

The Composition of Rabbits' Fat. L. Drumel. (*Bull. de l'Ass. belge des Chim.*, 1896, ix., 323.)—Rabbits' fat is white, or occasionally yellowish. Its odour is characteristic, and its consistency less than that of lard. The following constants were obtained by the author :

Density at 100° C.	0.861.
Melting-point	44.46° C.
Temperature of solidification	28.30° C.
Refractive index (Zeiss) at 40° C.	49.
Insoluble fatty acids	95.47 per cent.
Volatile fatty acid (Reichert-Meissl No.) for 5 grammes	2.64 $\frac{N}{10}$ NaOH.
Insoluble fatty acids	melting-point	48.50° C.
	solidification temperature	39.41°.
	refractive index at 40°	36.

C. A. M.

The Reaction for Sesame Oil. J. Wauters. (*Bull. de l'Ass. belge des Chim.*, 1896, ix., 257-280.)—Baudouin's test—the red colour produced by the action of hydrochloric acid containing sugar in solution—though giving good results when the sesame oil is present in sufficient quantity, is not very reliable when small amounts are in question. In 1893, Villavecchia and Fabris (*ANALYST*, xix. 47) studied the reaction, and found that the red colour was only produced when levulose, or substances producing levulose when acidified, were used. With other sugars, such as glucose, maltose, and galactose, the test failed. They came to the conclusion that the principal factor in the reaction was furfural, which is produced by the action of acid on certain kinds of sugar, notably levulose and saccharose, and in Baudouin's test they employed a solution of 2 grammes of furfural in 100 c.c. of alcohol instead of the sugar.

The author has examined this modification, and confirms its accuracy and delicacy. He proceeds in the following manner : 10 c.c. of hydrochloric acid are mixed with 0.1 c.c. of the alcoholic solution of furfural in a test-tube, 10 c.c. of the oil under examination added, and the whole allowed to stand. If sesame oil be present, the red colour is produced at the juncture of the two liquids. With 1 per cent. of sesame oil, the colour only appears after one or two minutes, but with 5 per cent. it is almost instantaneous.

With olive-oil, when fresh, no coloration is obtained, but with old and rancid samples there is a yellowish colour, which might possibly mask the addition of 1 per cent. of sesame oil, though 5 per cent. would be easily detected.

In examining butter, 10 c.c. are melted, filtered, and mixed with 10 c.c. of hydrochloric acid and 0.1 c.c. of furfural solution, the most suitable temperature being 40-45° C. Fresh butters give no coloration, and the addition of 0.25 per cent. of sesame oil is readily detected. With rancid butters a brownish-yellow colour is obtained, which might mask the addition of a very small amount of the oil.

This reaction may also be employed conversely to detect furfural in distillation products, the alcohol to be examined being shaken with 10 c.c. of hydrochloric acid and 10 cc. of sesame oil. The author has obtained in this way a reaction with alcohol containing as little as 1 part of furfural in 50,000.

C. A. M.

ORGANIC ANALYSIS.

On the Inversion of Sugar by Salts. J. H. Long. (*Jour. Amer. Chem. Soc.*, 1896, xviii. 120-130.)—It is well known that the specific rotation of cane-sugar solutions is decreased by the presence of many neutral salts, and K. Farnsteiner (*Ber.*, xxiii. 3570) has noted the connection between the molecular weights of the salts dissolved with the sugar and the amount of depression produced.

The author's experiments prove that temperature has a considerable influence, an inversion by means of ferrous iodide, which at the ordinary temperature requires months, being completed in less than two hours at 100° C. The rapidity of inversion is further increased by exposure to light.

In the experiments here summarized the solutions were heated in a small flask with a perforated rubber stopper, having a capillary glass tube in the perforation.

Ferrous Chloride.—Fifty grammes of cane sugar and 4.1 grammes of ferrous chloride in 100 c.c. Rotation in 100 mm. tube, 32.75°. Rotation in same tube after one hour at 100° C., -6.42°.

Ferrous Bromide.—Fifty grammes saccharose; 10 grammes of bromide in 100 c.c. Rotation = 32.25°. After one hour at 100° = -10.26° at 20° C.

Ferrous Sulphate.—Fifty grammes sugar, and 10 grammes of salt in 100 c.c. Rotation = 33°. After heating for an hour protected from the air = 18.20°.

Ferrous Ammonium Sulphate.—Fifty grammes sugar; 10 of the salt in 100 c.c. Rotation = 33.08°. After five hours at 71° C. = 27.20°. The solution, after standing in a stoppered bottle for four months exposed to light, decreased in rotatory power to 7.53°.

Manganous Chloride (4H₂O).—Fifty grammes of sugar and of the salt. Rotation = 32.88°. After four months in the light = 1.66°.

Manganous Sulphate (+4H₂O).—Fifty grammes sugar + 10 grammes of crystallized sulphate. Rotation = 33.16°. After one hour at 100° C., there was some decomposition. Rotation, about +7° in 100 mm. tube.

Zinc Sulphate.—Fifty grammes sugar, 10 grammes of the salt in 100 c.c. Rotation = 32.98. After forty-five minutes at 100° C. = 18.42°.

Potassium Aluminium Sulphate.—Sugar, 50 grammes; alum, 5 grammes in 100 c.c. Rotation = 33° . After one hour at 100° C. = -9.99° .

Lead Nitrate.—Sugar, 50 grammes; lead nitrate, 10 grammes in 100 c.c. Rotation = 33.13° . After one hour at 100° C. = -9.65° .

Lead Chloride.—Sugar, 50 grammes; lead chloride, 2 grammes in 100 c.c. Rotation = 23.5° . After heating = -7.53° .

Cadmium Chloride.—Sugar, 50 grammes; cadmium chloride, 8 grammes in 100 c.c. Rotation = 32.91° . After heating to 100° C. = -9.50° .

Mercuric Chloride.—Sugar, 50 grammes; mercuric chloride, 5 grammes. Rotation = 33.22° . On heating at 100° C., for some time there was a precipitate of mercurous chloride. Rotation of clear supernatant liquid = -10.80 . A second portion, heated for a shorter time till turbidity just commenced = -4.82° .

The above salts, though neutral, have a weak base as compared with the acid, and the author's further experiments seem to point to strong sugar-solutions being inverted by such salts, on account of the partial hydrolysis of the latter by the solvent. By diluting the solutions, the results were much more uniform, approaching those obtained with weak acids alone.

C. A. M.

On the Determination of Nitrites in Potable Water. A. H. Gill and H. A. Richardson. (*Jour. Amer. Chem. Soc.*, 1896, 21-23.)—In the examination of a large number of samples by means of Trommsdorff's iodo-zinc starch method and Griess' α -naphthylamine test discrepancies were often observed, especially in the case of peaty water. Upon decolorizing the waters the results agreed, proving that peaty matter interfered with the formation of starch iodide, and that unless nitrites were present in considerable quantity they could not be detected by Trommsdorff's test.

The decolorization was effected by shaking 250 c.c. of the water in the cold with "milk of alumina," allowing to settle, and filtering through a filter which had been washed free from nitrites. The "milk of alumina" was prepared by precipitating a boiling solution of 125 grammes of potash alum per litre with ammonia, allowing the precipitate to settle, and washing by decantation.

C. A. M.

Examination of Oil of Bergamot. A. Bornträger. (*Zeit. anal. Chem.*, 1896, xxxv. 35-38.)—Bergamot oil owes its characteristic odour to the presence of linalol acetate, $C_{10}H_{17}O.C_2H_3O$, the amount of which appears to be fairly constant. From the examination of a large number of specimens, Schimmel and Co. gave this amount at 34 to 43 per cent., while the author found from 37.6 to 39.9 per cent. The determination of this constituent is, therefore, a valuable means of detecting adulteration with oil of turpentine. From 1 to 2 grammes of the ethereal oil are boiled with 10 to 20 c.c. of semi-normal potash under a reflux condenser for one to two hours, and titrated back with semi-normal sulphuric acid, phenolphthalein being used as indicator. The amount of free acid in the bergamot oil is, as a rule, so trifling that it may be neglected.

On evaporating bergamot oil on the water-bath, the residue should not be more than 6 per cent. of the weight taken. In five specimens known to be genuine, the

author found from 4.2 to 5.7 per cent. From 1 to 2 per cent. of saponifiable matter is contained in this residue, and this should be deducted from the amount of linalol acetate previously determined. A large amount of saponifiable matter in the residue would indicate adulteration with fatty oil.

The addition of colophony would be shown by the residue being too high, and by the amount of free acids (abietic, etc.) being more than normal. C. A. M.

The Qualitative Examination of Acetanilide. C. Platt. (*Jour. Amer. Chem. Soc.*, 1896, xviii. 142-146.)

Physical Characteristics.—Melts at 113° to 114° C. At 15° C. is soluble in about 190 parts of water and in 5 parts of alcohol.

Concentrated Nitric Acid.—Readily soluble; solution colourless when cold, but changes to yellow and red on warming. The cold solution gradually changes to red on standing, with formation of red acicular crystals. The red solution, on warming, gives off the odour of nitro-benzene.

Concentrated Sulphuric Acid.—Colourless solution, unaffected by boiling. On long standing the solution changes to pink or brown.

Sulphuric Acid and Potassium Bichromate.—Dark-green solution.

Hydrochloric Acid.—Readily soluble when warmed.

Hydrochloric Acid and Potassium Permanganate.—Olive-green colour.

Hydrochloric Acid and Chromic Acid.—On adding a weak solution of chromic acid to the hydrochloric acid solution, a green coloration is obtained. Potassium hydrate gives a blue precipitate in this solution.

Hydrochloric Acid and Bromine.—Bromine water added to hydrochloric acid solution gives a heavy yellow-to-white precipitate of the mono-brom derivative of aniline.

Hydrochloric Acid and Chlorine.—Dark-blue coloration, which afterwards fades.

Potassium Hydrate.—Odour of aniline on heating.

Sodium Nitrite and Sulphuric Acid.—The powder mixed with sodium nitrite, and sprinkled on concentrated sulphuric acid, gives a red colour.

Ferric Chloride.—Produces no change when added to a cold saturated aqueous solution.

Zinc Chloride.—Heated to 270° with an equal weight of acetanilide produces first ortho-amido-acetophenone (a yellow oil with high boiling-point) in small amount, and then flavaniline ($C_6H_4N_2$), a yellow substance with green fluorescence, a derivative of quinolin. The test of boiling acetanilide with zinc chloride, aniline, and acetic acid is considered untrustworthy by the author, who asserts that the reagents alone will produce amido-aceto-phenone ($C_6H_4NH_2C_2H_3O$).

Plugge's Reagent.—Boil the acetanilide with water, filter, boil with potassium nitrite and dilute nitric acid. Mix with Plugge's reagent (a solution of mercurous nitrite with a little nitrous acid), and again boil. A deep-red colour is produced.

The foregoing tests readily distinguish acetanilide from antipyrin and phenacetin (cf. ANALYST, xxi. 69). C. A. M.

INORGANIC ANALYSIS.

Yellow Tint of Zinc-white. F. Fucks and F. Schiff. (*Oest. Z. Berg. u. Hütten-w.*, 1896, 29; through *Chem. Zeit. Rep.*, 1896, 42.)—The authors find that the yellow tint occasionally met with in zinc-white, and which has been hitherto attributed to the presence of ferric oxide, is due to minute traces of cadmium sulphide. Artificial mixtures prepared with cadmium and ferric oxide showed that the alteration in colour produced by these bodies was scarcely perceptible.

W. J. S.

Estimation of Manganese in Pig Iron. F. Ulzer and J. Brüll. (*Mittheil. techn. Gew.-Mus.*, Vienna, 1895, v., 312; through *Chem. Zeit. Rep.*, 1896, 36.)—The solution, freed from iron by means of zinc oxide (Volhard's process, *Fresenius*, ii., 443), and containing about 0.1 gramme of manganese, is treated with 20 c.c. of 5 per cent. hydrogen peroxide, caustic soda added as long as a precipitate falls, boiled, cooled, and oxalic acid of known strength introduced. The precipitate is dissolved with pure dilute nitric acid, the solution heated to the boil, and the excess of oxalic acid titrated. The composition of the manganese precipitate may be taken as $5\text{MnO}_2\text{Mn}_2\text{O}_7$. To dissolve the original sample, 10 parts by volume of strong nitric acid, 2 parts of sulphuric acid, and 10 parts of water are employed, about 10 c.c. of hydrochloric acid being added during the evaporation.

F. H. L.

Analysis of Persulphates. F. Ulzer. (*Mittheil. techn. Gew.-Mus.*, Vienna, 1895, v., 310; through *Chem. Zeit. Rep.*, 1896, 36.)—To determine the active oxygen, 0.3 gramme of the sample is heated with 1 to 1.5 gramme of iron ammonium sulphate for half an hour in a current of carbon dioxide, and the unoxidized iron estimated with permanganate. The results agree within 0.2 per cent. In one particular specimen of the ammonium salt, 3.20 per cent. of oxygen was found, equivalent to 45.61 per cent. of ammonium persulphate. For the total sulphuric acid, a fairly strong solution was heated with hydrochloric acid for some time, then precipitated with barium chloride, the yield being 70.01 per cent. The ammonia was 14.01 per cent., and the fixed residue 0.61 per cent. On titrating the aqueous solution with the aid of methyl orange, 18.93 per cent. of sulphuric acid was found. From these figures the composition of the substance works out as follows:

	Per Cent.
Ammonium persulphate	45.61
" bisulphate	48.77
Fixed residue	0.61
Moisture	5.01

F. H. L.

Silica a Cause of Scale in Boilers. A. Reichard. (*Chem. Zeit.*, 1896, p. 65.)—The following instance shows that it is unsafe to judge of the fitness of a water for feeding boilers merely from the amount of calcium and magnesium salts which it contains. A water yielding a residue on incineration of 8.60 per 100,000, and con-

taining lime 2.08, magnesia 0.22, silica 2.6, alkalies (weighed as chlorides) 2.35 parts, gave serious trouble in the formation of incrustation. The scale was found to consist chiefly of silica and lime. Weathered granite in the formation from whence the water was obtained was the cause of the large amount of silica and alkalies.

W. J. S.

Determination of Gold in Pottery Enamel. M. Schirmer. (*Ann. de Chim. Analyt.*, i., 47.)—Owing to the presence of compounds, such as resinate of silver added to dilute or cheapen the enamel, an addition which may amount when fine gold is used to 8 per cent. of metallic silver without exerting too great an influence on the colour, and especially to the mineral flux—most frequently a fusible salt of bismuth—which is always added to the enamel for the purpose of fixing the gold on the glass or porcelain surface, a simple incineration will not be sufficient to determine the gold. The following method is recommended as giving satisfactory results:

Ten grammes of the "liquid gold" to be tested is weighed rapidly in a large porcelain crucible, and evaporated on the sand-bath at a low temperature. The residue is then incinerated at the lowest possible temperature, since if the glaze on the crucible be softened some of the gold may be absorbed. The residue is taken up with cold aqua regia, and the solution, after dilution with water, is decanted on to a filter, and the residue treated once or twice with warm acid of the same strength. Finally, all the filtrates and washings are united, and the gold thrown down by ferrous sulphate in the usual manner.

Should the inside of the crucible exhibit a violet or rosy coloration, indicating the absorption of gold by the glaze, it must be finely powdered and treated with cold aqua regia, the solution and washings being joined to the others; but this complication will not arise if the incineration has been properly performed.

C. S.

CORRESPONDENCE.

TROPÆOLINS IN MILK, ETC.

To the Editors of THE ANALYST.

DEAR SIR,—In order to detect these dyes in milk it is of the greatest importance that the sample should be tested directly it is received by the analyst, as the destruction of tropæolin, probably by the action of some ferment, commences very early in the decomposition of the milk.

I have observed that 10 c.c. of fresh milk (in winter) will consume in three days nearly 0.2 c.c. of solution of tropæolin (1 in 1,000), this being enough of the dye to impart a very decided yellow or orange colour to the milk, and that the destruction afterwards proceeds much more rapidly.

Boiling, after the action is well advanced, temporarily arrests for many days the destruction of the dye, but previous boiling of fresh milk only very slightly delays the commencement of the process.

Yours faithfully,

LESTER REED, F.I.C., F.C.S.

CROYDON, February 25, 1896.

THE ANALYST.

JUNE, 1896.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, May 6, in the Chemical Society's Rooms, Burlington House, at 8 o'clock, the President (Dr. Stevenson) occupying the chair. The minutes of the previous meeting were read and confirmed.

THE PRESIDENT announced that Mr. Hudson E. Kearley, M.P., had been unanimously recommended by the Council for election as an honorary member of the Society. Mr. Kearley had rendered very essential services to the Society in his capacity as a member of the Select Committee of the House of Commons on Food Products Adulteration, and it would be a very graceful act on the part of the Society to elect him an honorary member in recognition of those services.

The following papers were read: "The Composition of Human Fat," by C. A. Mitchell, B.A.; "Note on an Incrustation found in Hot-water Pipes," by J. Augustus Voelcker, M.A., B.Sc., Ph.D.; "The Examination of Commercial Milk Sugar," by H. Droop Richmond; "Note on 'drawn' or exhausted Caraways," by Bernard Dyer, D.Sc., and J. F. H. Gilbard.

THE BACTERIOLOGICAL EXAMINATION OF WATER FOR THE TYPHOID BACILLUS.

BY T. H. PEARMAIN AND C. G. MOOR, M.A.

(Read at the Meeting, April 1, 1896.)

(Concluded from p. 122.)

THE ISOLATION OF THE TYPHOID BACILLUS FROM WATER.

IN waters that have been very copiously contaminated with sewage there is no great difficulty in detecting the typhoid or colon bacillus if present, but it is necessary to bear in mind that usually when drinking-water has suffered sewage pollution the quantity of the polluting matter is relatively very minute when compared with the great bulk of the water-supply. The contamination of water by sewage is, moreover, in the majority of cases of an intermittent nature.

When such waters are examined, as large a quantity as 1 cubic centimetre or more of the water may be "plate cultured," and even then it is easy to miss the colon bacillus, not to speak of the typhoid bacillus.

In order to isolate the *B. typhosus* suspected to be present in a sample of water,

it is necessary to submit a large volume of the water to examination. This object is attained by concentrating the bacterial contents of the water by passing 1,000 to 3,000 c.c. or more of the sample through a small sterile Pasteur-Chamberland, or, better for practical purposes, a small Berkefeld filter. By this treatment all the bacteria in the water are retained on the outer surface of the filter. The particulate matter thus retained is then brushed off the outer coating of the filter with a sterile brush into about 20 c.c. of sterile distilled water. One c.c. of this, containing the particulate matter from 50 to 150 c.c. of the original water, is then immediately submitted to plate-culture by one of the undermentioned methods to isolate the colon bacillus and also the typhoid bacillus if present.

(1) *Inhibition by Means of Phenol.*

The *B. typhosus* and the *B. coli communis* are among the limited number of micro-organisms which will grow in the presence of small quantities of phenol, which addition retards, or inhibits the common water bacteria, such as the *B. fluorescens liquefaciens*, *Proteus vulgaris*, *B. mesentericus*, etc., the presence of which would liquefy the gelatin, and by their rapid growth would annihilate the *B. typhosus* if present. The presence of a small quantity of phenol does not in any way interfere with the growth of the *B. typhosus* or the *B. coli*, but exhibits a marked inhibitory effect upon the common water bacteria, and, by the retardation and suppression of these, the colonies of the *B. typhosus* and the *B. coli* have time and opportunity to appear.

Phenol appears to have been first used for this purpose by Chantemesse and Widal,* who employed nutrient gelatin containing 0.25 per cent. of phenol. Thoinot,† a little later, inhibited the growth of organisms, other than the typhoid and colon bacillus, by adding 0.25 per cent. of phenol to the water under examination, which was then incubated at blood-heat and the water afterwards plate-cultured.

As pointed out by Holz, and confirmed by Dunbar, the above authors use a percentage of phenol which altogether prevents the growth of the *B. typhosus*. Dunbar states that 0.12 per cent. of phenol greatly interferes with the growth of the typhoid bacillus, while in the presence of 0.14 per cent. it will not develop at all. He further states that in the presence of small quantities of phenol the colon bacillus presents stronger resemblances to the typhoid bacillus than usual.

To ascertain if the resisting power of cultures of the *B. typhosus* to phenol differed, we tried the following series of experiments on different cultures of the organism, using varying percentages of phenol, with the following results :

Percentage of Phenol.				0.05.	0.10.	0.20.	0.30.
<i>B. typhosus</i>	(a)	+	-	-	-
"	(b)	+	+	-	-
"	(c)	+	+	+	-
"	(d)	+	+	-	-
<i>B. coli communis</i>		+	+	+	+

Thus, it is seen that the resisting power of the *B. typhosus* to phenol varies with different cultures. The sample marked (a), which was freshly isolated from the

* *Gazette des Hôpitaux*, 1887, p. 202.

† *Ibid.*, 1887, p. 348.

dejecta from a typhoid case, had less resisting power than other samples which had been sub-cultured through many generations.

Parietti proposed the use of broth containing both phenol and hydrochloric acid to eliminate the common water organisms. He takes advantage of the fact that the typhoid and colon bacillus will grow in a slightly acid medium, whereas the majority of other organisms will not.

Parietti's method is as follows: The following solution is prepared: Five grammes of phenol and 4 grammes of pure hydrochloric acid are added to 100 c.c. of distilled water. From 0.1 to 0.3 c.c. of this solution is added to a series of test-tubes containing 10 c.c. of sterile nutrient broth (= 0.05 to 0.15 per cent. of phenol). The tubes are then incubated at blood-heat for twenty-four hours, to destroy any stray organisms that may have gained access to the tubes. From 0.1 to 0.5 of a c.c. of the water under examination is then added to the tubes, the contents well mixed, and the tubes again returned to the incubator. If, after twenty-four hours' incubating at blood-heat, any of the tubes appear to be turbid, they are submitted to ordinary plate-cultivation and the resulting colonies carefully examined in sub-cultures. Frankland states that when only a few typhoid bacilli are present, the incubation must be prolonged for forty-eight or even seventy-two hours.

We have found the above method to be a very reliable one, although somewhat tedious. In practice, however, we prefer to use simple carbolized gelatin containing 0.05 per cent. of phenol. This quantity is quite sufficient to restrain the growth of liquefying organisms, and, moreover, with this quantity there is no danger of losing the typhoid bacillus if it is present.

(2) *Elsner's Method.*

Dr. Elsner, of Berlin, has recently published* the results of an investigation made to ascertain the possibility of an early recognition of enteric fever by the bacteriological examination of the stools. He has been able to recognise the Eberth-Gaffky bacillus in some cases in as short a time as forty-eight hours. Dr. Elsner went over the existing methods for the separation of the *B. typhosus* and *coli*, with no better results than have previously been obtained. In all cases but one he found that either persistent organisms other than those sought to be isolated would grow to a sufficient extent to spoil the plate (e.g., *B. proteus* or *ramosus*), or else the *B. coli* would develop to an extent capable of preventing the recognition of the typhoid bacillus. The exception was slightly acid potato-gelatin, containing 1 per cent. of iodide of potassium. The process recommended is to boil potato-decoction (500 grammes to 1 litre of water) with 10 per cent. of gelatin. Sufficient of a 2 per cent. solution of sodium hydrate is added till only a faint acidity remains, litmus being used as indicator.

Elsner found that the *B. proteus* and *ramosus*, which always grow on carbolized gelatin, either never occurred on this medium, or were rapidly overgrown by the colon bacillus. The *B. coli* grew in twenty-four hours, presenting the usual appearance of that organism on acid media; the *B. typhosus* was scarcely visible in twenty-four hours, but in forty-eight hours appeared in small, shining, very finely-

* *Zeitschr. f. Hyg.*, xxi. 1.

granulated colonies like little drops of water, which contrasted strongly with the larger coarsely-granulated, brownish colonies of the colon bacillus. The *B. coli* only acquired the appearance of the typhoid colonies when a great number of the organisms were present, and many, therefore, grew without finding room for their proper development. In plates made with weaker inoculations, it is impossible to mistake one bacillus for the other. We have used this method with satisfactory results. The colonies of the *B. typhosus* appear more quickly on this medium than on carbol-gelatin, but otherwise this appears to be the only advantage it possesses.

A number of other methods for the isolation of the *B. typhosus* and *coli* have been proposed by different investigators. Uffelmann has suggested the addition of 0.1 per cent. of citric acid to the nutrient gelatin, to restrain the growth of the liquefying organisms. Dunbar has found, however, that the amount of citric acid prescribed by Uffelmann is in excess of what the typhoid bacilli are capable of withstanding. He found that in many cases, whilst the colon bacillus developed, the growth of the *B. typhosus* was restrained.

Holz has used faintly acid potato-juice, thickened with 10 per cent. of gelatin, with or without the addition of 0.05 per cent. of phenol, with satisfactory results. This method is practically the same as Elsner's, except that the potassium iodide is replaced by phenol. Gasser, Holz, Lyonnet, and others have suggested the use of various media tinted with fuchsine and other aniline dyes. The typhoid and colon bacilli are stated to decolorize, or to cause other changes, in such media where the growth of the colonies occurs, whereas other organisms do not possess this property. We find that but little reliance is to be placed on these appearances, as we have found other organisms give the same characteristics as the typhoid and colon bacilli.

After a considerable experience in the use of the above methods, we find that the best and most reliable processes to be employed for the isolation of the typhoid bacillus from water are the use of carbolized gelatin (0.05 per cent.), Elsner's method, and Parietti's acid carbolized broth. As soon as the colonies which develop on the carbolized or potato-gelatin become sufficiently advanced they are examined with a lens, and any suspicious colonies are carefully sub-cultured into faintly alkaline sterile milk-tubes, which are then incubated at 37° C. for thirty-six hours. The milk-tubes are then examined, and any that have become coagulated are rejected, as certainly not typhoid.

From the tubes that have not coagulated the following sub-cultures are prepared: (a) Gelatin "streak" culture; (b) gelatin "shake" culture; (c) broth culture.

The gelatin cultures are kept for three days at a temperature of from 18° to 20° C. The broth-tubes are incubated at blood-heat for the same length of time, and then tested by the indol reaction.

With reference to the general question of the bacteriological examination of drinking-water, much information as to the character of a water is gained by incubating a small quantity of the sample at blood-heat for twenty-four hours. The number of organisms is then ascertained by an ordinary gelatin plate culture. The

number of organisms so found is compared with the number of organisms found by a direct gelatin plate culture which is made on the water immediately upon the receipt of the sample. If a sample of water is polluted with sewage, a great increase in the number of the organisms will be found to have taken place as the result of the incubation. All the organisms normally present in faeces grow and multiply vigorously at blood-heat, whereas this temperature is fatal to the majority of the common water bacteria; therefore a corresponding decrease in the number of the organisms will be found to have taken place in a pure water.

A more convenient plan is to prepare an agar-agar plate culture with a fraction of a cubic centimetre of the water. The resulting-plate is incubated at blood-heat for thirty-six hours. This method is the most satisfactory, as it has the advantage that the actual number of the micro-organisms that will grow at blood-heat is ascertained.

The following examples show the value of these two methods :

	Polluted Surface-well Waters.			Waters of Average Quality.	
	(a)	(b)	(c)	(d)	(e)
Approximate number of organisms per c.c. in the original water, as determined by a gelatin plate culture	800	1,050	1,400	180	270
Number of organisms per c.c. appearing on an agar-agar plate colony, after incubating at blood-heat for twenty-four hours	220	180	350	10	5
Approximate number of organisms per c.c., after incubating the water at blood-heat, the organisms then being determined by an agar-agar plate... ..	800,000	uncountable.	uncountable.	—	—

The majority of the organisms from a polluted water which grow at blood-heat will be found on sub-culturing to be the colon bacillus. The presence of the *B. coli communis* in small numbers can hardly be considered as good evidence of sewage-pollution, but when it is found in large numbers it is fair to conclude this to be the case.

The colon bacillus is spoken of by Klein* "as a certain index of faecal pollution."

The recent researches of Dr. A. A. Kanthack, however, show that the *Bacillus coli* is much more widely distributed than was formerly supposed, being found by him in pure water, saliva, dust, etc., so that the generally prevailing idea that its presence necessarily signifies excretal pollution is erroneous. The widespread distribution of the *B. coli communis* has, however, long been known to bacteriologists, and it is comparatively rare to find it absent from waters of high degree of purity that have been exposed to the air.

* The 23rd Annual Report of the Local Government Board.—Supplement containing the Medical Officer's Report, p. 67.

As pointed out by Dr. Thresh* in a recent paper before this Society, all surface-waters contain large numbers of micro-organisms, and waters from deep-seated springs are almost sterile. When such pure waters are allowed to stand for a few days, however, the number of organisms increase enormously. Frankland states that a pure water containing, say, five organisms per cubic centimetre when freshly drawn, may, even if kept in a sterile flask free from aerial contamination, contain after a few days, perhaps, 500,000 in the same volume—or, in other words, as many as are found in slightly-diluted sewage. He also points out, however, that whilst in sewage the number of organisms only gradually diminishes, in these pure waters “after the rapid increase in numbers follows a correspondingly rapid decline, so that the numbers again fall below those found in impure surface-waters.” The above facts must be constantly before one when interpreting the results yielded by the bacteriological examination of a sample of water.

Messrs. Laws and Andrewes† failed, after a most prolonged investigation, to find the typhoid bacillus in the London sewage from the Barking and Crossness outfalls, but they found it present, as would be expected, in the sewage from the Homerton Fever Hospital.

With respect to the question of the detection of the typhoid bacillus in water, we are satisfied that the Eberth-Gaffky bacillus can be, and has actually been, detected and isolated from water, though some of the cases in which it has been reported may rest upon insufficient evidence. We would, however, consider that the discovery of any of the pseudo-typhoid organisms, such as have been already mentioned, should lead to as decided a condemnation of the water as though an organism possessing the precise morphological and cultural characters of the Eberth-Gaffky bacillus were isolated.

While we would not agree with those who would regard the bacteriological examination as useless, we still further dissent from the view—if, indeed, it is seriously held by any—that the biological examination can in the smallest degree supplant the chemical analysis of water, which, on account of the valuable data it yields, must always remain an integral part of the examination of potable water.

The most enthusiastic bacteriologist cannot deny that the specific organism may have been present in a given water-supply a week ago, and at the time of examination have disappeared. The incubation period of enteric fever is about fourteen days; so that if a sample of drinking-water were sent for examination when the disease declared itself, it might easily be three weeks since the conveyance of the infection, and during this time the Eberth-Gaffky bacillus may have been annihilated by the common water bacteria.

Therefore, to say that a given water was safe because no specific organism was demonstrable, and to ignore the information that a chemical analysis might yield, would be entirely illogical.

DISCUSSION.

Mr. JOSEPH LUNT, B.Sc., said he had listened to the paper with very great interest. The matter was a most difficult one, and one to which he thought Public

* ANALYST, 1895, p. 99.

† *Report on the Results of the Investigations on the Micro-Organisms of Sewage*, presented to the London County Council, December, 1894.

Analysts ought to pay a great deal of attention. He would have liked to hear the middle portion of the paper, which dealt with the methods used for separating the typhoid bacillus from water in presence of the *Bacillus coli communis*. It was very important to distinguish between the typhoid bacillus and the pseudo-typhoid bacilli, which were apt to occur in water. He would not like to go so far as the authors of the paper, in saying that all waters ought to be condemned which contain these pseudo forms resembling typhoid, because he was afraid that if such a course were followed, there would be condemned a great many good waters which were in constant use without harmful results. Only a few days ago he had had a sample of water for examination in the Water Laboratory of the British Institute of Preventive Medicine, which gave a bacillus which resembled the typhoid bacillus most closely, but which was not identical with it, although other indications seemed to show that the water should not be condemned. It seemed to him that such points as this required a very great deal of investigation before a satisfactory conclusion could be come to. To condemn all waters which contained typhoid-like organisms would, he was afraid, be going rather too far.

Dr. HEWLETT said that he had not had so much experience in this direction as Mr. Lunt. Certainly the question of organisms resembling the typhoid bacillus—the so-called “pseudo-typhoid bacilli”—made the matter extremely difficult to deal with, and perhaps there might be some danger in anyone taking up the bacteriological examination of water, unless he had spent a considerable amount of time in studying the methods and in differentiating these organisms. In some cases the differentiation might be very difficult, and might require a larger amount of time than most Public Analysts would be likely to have at their disposal. He quite agreed with the authors that the chemical and biological examinations of water should go hand-in-hand. He did not think anyone, in the present state of knowledge, would rely upon bacteriological examination alone. A point had been raised with regard to the identity of the *Bacillus coli communis* and of the typhoid bacillus from the resemblance of the action of their metabolic products. He did not think this was a matter of great importance, because many organisms which were totally different would protect against other organisms. The *Bacillus prodigioides*, for example, would protect against the cholera bacillus, two organisms which could not be mistaken for each other in any way. What he would have liked to have heard was of some method for separating the *Bacillus coli communis* when this and typhoid were present together.

The PRESIDENT said he had listened with great interest to this paper, much of which was perhaps foreign to the ordinary work of the Public Analyst, viz., the very interesting résumé which had been given of what had been done with regard to ascertaining the pathogenic or other characteristics of the typhoid and other organisms. What was perhaps most specially interesting to Public Analysts was the differentiation between the true typhoid bacillus and the *Bacillus coli* and other typhoid-like organisms. The subject was one in which he himself had taken a great interest during the past year in connection with the investigations with regard to the water-supply of London. The great difficulty arose after the organisms had been reduced by cultivation at high temperatures to, say, typhoid and coli. The difficulty was not so great when there were large numbers of typhoid organisms present, or reason to

suspect them; but when search had to be made for the typhoid organism in very small numerical proportions, in the presence of large numbers of *Bacillus coli*, the difficulty became very great. From his own experience, one could not rely upon one single indication without confirmation by several well-known tests, and this, if he had understood the paper aright, was the conclusion which the authors had come to.

NOTE ON THE ESTIMATION OF FORMIC ALDEHYDE.

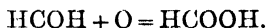
By HARRY M. SMITH.

(Read at the Meeting, April 1, 1896.)

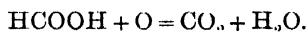
ACTING on the suggestion of Dr. Stevenson, I have made experiments as to the conditions affecting the oxidation of formic aldehyde into formic acid, with the following results:

When formic aldehyde is treated with excess of an alkaline solution of potassium permanganate in the cold, it is completely oxidized into formic acid. The absence of formic aldehyde after oxidation is proved by filtering, removing the excess of permanganate by means of oxalic acid in the presence of sulphuric acid, and treating the slightly acid solution with Schiff's reagent. The presence of formic acid is proved by its reducing action on ammoniacal silver nitrate and on mercuric chloride. If, however, formic aldehyde is *boiled* with excess of an alkaline solution of potassium permanganate, it is oxidized to carbon dioxide and water. The oxidation, therefore, takes place in two stages, thus:

1st stage: In the cold—



2nd stage: On boiling—



By careful attention to temperature these reactions can be made strictly quantitative.

The reagents required are:

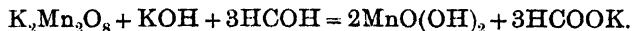
1. *A standard solution of potassium permanganate.* This is prepared by dissolving 5.260 grammes potassium permanganate in 1,000 c.c. of water. When used in alkaline solutions,

1 c.c. = 0.00080	gramme oxygen.
1 c.c. = 0.00115	„ formic acid (boiling).
1 c.c. = 0.00075	„ formic aldehyde (boiling).
1 c.c. = 0.00150	„ formic aldehyde (cold).

2. *A solution of potassium hydroxide.* This is prepared by dissolving 50 grammes of stick potash in water and making the solution up to 100 c.c.

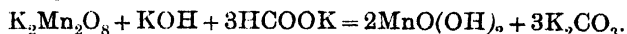
As a preliminary step, a measured quantity of the sample of formic aldehyde is placed in a porcelain dish, and potassium hydroxide solution added, so that the solution shall contain at least 10 per cent. of alkali. The permanganate solution is then run in slowly from a burette. At first a fine green coloration is obtained from the formation of potassium manganate, but this rapidly turns to a reddish-brown,

especially if the solution is gently warmed. The addition of permanganate is continued, with constant stirring, preferably with a small thermometer, until the green colour disappears rather slowly. The solution is then warmed to 30° C., as this temperature aids the separation of the precipitate of hydrated manganese peroxide, and the colour of the supernatant liquid can then be observed. The addition of permanganate is continued, 0.5 c.c. at a time, until an olive-green coloration, permanent for about fifteen to twenty seconds, is obtained, this being about the time necessary for the precipitate to subside. This is the end of the first stage of the reaction, which may be represented by the following equation :



It should be noted that the addition of permanganate in slight excess will not increase the colour of the supernatant liquid, but the precipitate appears much darker for a time.

The quantity of permanganate used should be noted, and a further quantity equal to the first less 2 c.c. is at once run in, and the solution raised to the boil. The heating is stopped, and the precipitate allowed to settle. There should be no green coloration, but on the further addition of permanganate solution, four or five drops at a time, raising to the boil between each addition, an emerald-green colour, permanent for at least ten minutes, is finally obtained. This indicates the end of the second stage, the equation being :



Having obtained from this experiment a rough estimate of the dilution required, which should be such that the colour of the supernatant liquid can be easily observed, a second experiment should be made, in which nearly the whole of the permanganate solution required for the completion of the first stage of the reaction should be run in at once. The solution is then heated to a temperature not exceeding 30° C., and the reaction completed as quickly as possible in the manner described above.

If the temperature be raised above 30° C., or if time is lost in determining the end point of the reaction, as indicated by the green colour of the supernatant liquid, oxidation of the formic acid occurs.

The second stage of the reaction is completed as described in the first experiment, and requires no special precautions.

It must be remembered, however, that solutions of alkaline manganates are gradually decomposed by carbonic acid, and therefore prolonged heating must be avoided. The presence of not less than 10 per cent. of alkali is advisable in order that the green colour of the manganate may be sufficiently pronounced to admit of a delicate end point.

The acidity of the sample of formic aldehyde should be ascertained, and a deduction made, if necessary, for the formic acid present. But the largest amount of formic acid likely to be present in a sample of commercial formalin is so small that its influence on the results obtained by the method described is inappreciable.

It is evident that the second stage of this method of analysis may be applied to the estimation of formic acid.

H. C. Jones (*Amer. Chem. Jour.*, 1895, xvii.) describes a somewhat similar process to this for the estimation of formic acid (see *abs ract*, ANALYST, xx., 205).

The following table gives the results of analyses, by the permanganate process, of dilute solutions of a sample of formalin. The figures in the second column are based on an analysis of the sample by the ordinary method, depending on the conversion of formic aldehyde by the action of ammonia into hexamethylene tetramine :

ANALYSES OF SOLUTIONS OF FORMALIN.

1. C.c. of Dilute Solution taken.	2. Gramme of Formic Aldehyde present.	3. Gramme of Formic Aldehyde found.	
		1st Stage.	2nd Stage.
1	0.024	0.024	0.025
4	0.032	0.034	0.034
10	0.080	0.087	Not determined
10	0.055	0.058	0.058
20	0.009	0.010	0.009
30	0.003	Not determinable	0.002
20	0.0009	" "	0.0006
50	0.0045	" "	0.0028
25	0.0022	" "	0.0015

The experiments hitherto described apply only to aqueous solutions of formic aldehyde, but I am making experiments with a view to the application of the method to the estimation of formalin in milk and other articles of food.

The results of these experiments I hope to lay before the Society on some future occasion, but it may be useful to state here that I have found that the difficulty met with in the distillation of milk may be entirely obviated by passing a current of steam through the liquid instead of heating it directly.

In conclusion, I have to thank Dr. Stevenson, and also my friend Mr. Norman Leonard, for kind assistance given me in the work described in this paper.

DISCUSSION.

Mr. HEHNER remarked that if a sample of milk containing formalin were distilled and tested for formaldehyde, it would not be possible to get the same depth of reaction as would be obtained with a sample of water containing the same quantity of formalin. Further, if this test were applied to any substance which had undergone fermentation, and in which traces of alcohol had consequently been formed such traces of alcohol would also cause a reaction, and would be included as formaldehyde in the result.

Dr. RIDEAL said it seemed to him that the small quantity of formalin which had to be dealt with when it was used as a preservative would not lend itself to exact estimation by means of titration with permanganate.

Mr. CASSAL said it was certainly very doubtful that formalin estimated in the distillate from milk could be taken as approximately representing that which was originally added to the milk. The action of formalin upon the constituents of milk—upon the "casein," in fact, as upon fibrin, albumin, and so forth—was evidently of a

very decided character, and the formalin originally added could hardly be recovered by a distillation process.

Mr. BEVAN said he did not think there was any doubt at all about the first point to which Mr. Hehner had referred. The amount of formalin recovered was by no means representative of the amount added, a fact which he believed to have been shown long before he himself read some notes on the action of formalin on milk. The fact that the weight of the total solids could be increased almost in proportion to the amount of formalin added was evidence that there was a combination, and it was clearly not possible to get back all the formaldehyde by distillation.

Mr. CASSAL said that the increase in the weight of the total solids was presumably due to the permanent effect produced by formalin on milk, but he did not know if Mr. Bevan had carried his experiments further, and had dried the total solids to constant weight. If formalin was present, it was necessary to dry for a very long time, but it was possible to get a very near approach to the actual total solids.

Dr. RIDEAL thought there might be some doubt as to whether the increase of the weight of the total solids was due to an addition of formaldehyde to the milk solids which remained after drying. It was conceivable that the formalin caused a combination of water with the milk-solids, and that the increase was not due to a direct combination with the formaldehyde. He believed that some of these compounds dissociated at boiling-point, the formaldehyde coming off again. It was, however, perfectly true that the distillate from milk containing formaldehyde would not give so much coloration as a water solution containing the same proportion of formaldehyde.

Mr. SMITH, referring to Mr. Hehner's remarks, said it was quite true that alcohol and many other organic substances, if present, would interfere with the titration, but that the oxidation of the aldehyde in the cold was so instantaneous that it might be possible to make an estimation in the presence of less easily oxidizable substances. With regard to the distillation of milk, he had found that with, say, 100 c.c. of milk containing a certain amount of formaldehyde, distilled in a current of steam, about 300 c.c. of distillate might be collected, with the aldehyde still coming over. The aldehyde, in fact, distils over very slowly, and this is also the case with aqueous solutions.

OFFICIAL METHODS FOR THE ANALYSIS OF FERTILIZERS, ISSUED
BY THE GERMAN MANURE MANUFACTURERS' ASSOCIATION,
HARZBURG, MAY 28, 1895.

CONTRIBUTED BY H. H. B. SHEPHERD, F.I.C., ANGLO-CONTINENTAL GUANO WORKS,
LONDON.

(Continued from p. 132.)

B. *Determination of the Citrate-soluble Phosphoric Acid by Wagner's Method* (Chem. Zeit., No. 63, 1895).—I. *Preparation of the Solutions.*—(1) *Concentrated Ammonium Citrate Solution.*—This solution must contain 150 grammes of pure crystallized citric acid and 23 grammes of ammoniacal nitrogen (27.93 grammes NH_3).

per litre. The citric acid can be added by weight and the ammonia adjusted by analysis.

We have prepared, for example, 10 litres of this solution as follows: 1,500 grammes of citric acid were dissolved in about 2 litres of water, with the addition of 3,500 c.c. of an 8 per cent. ammonia solution. After cooling, the solution was made up to exactly 8 litres with water, and 25 c.c. of this solution were taken and diluted to 250 c.c. Twenty-five c.c. of this diluted solution were then used for an ammonia determination. Two hundred c.c. of water and 3 grammes of calcined magnesia were added, and the ammonia distilled over into 40 c.c. $\frac{N}{2}$ sulphuric acid. The ammoniacal nitrogen found corresponded to 20.0 c.c. $\frac{N}{2}$ soda solution; consequently the 8 litres of concentrated citrate solution contained $\frac{20.0 \times 0.0035}{2.5} \times 8000 = 224.0$ grammes

ammoniacal nitrogen. In order, therefore, to produce 10 litres of solution containing 1,500 grammes citric acid and 230 grammes ammoniacal nitrogen, it was necessary to add to the 8 litres a further 2 litres containing $230 - 224 = 6$ grammes ammoniacal nitrogen, or 7.3 grammes ammonia, or 91 grammes of 8 per cent. ammonia, or 94 c.c. ammonia of 0.967 specific gravity.

(2) *Diluted Ammonium Citrate Solution*.—Two volumes of concentrated ammonium citrate solution are diluted with three volumes of distilled water.

(3) *Molybdenum Solution*.—This solution may be prepared in either of the following ways:

(a) One hundred and twenty-five grammes of molybdic acid are placed in a litre flask, and dissolved by the addition of about 300 c.c. of an 8 per cent. solution of ammonia. Four hundred grammes of ammonium nitrate are then added, and the solution made up to the mark with water. This is then mixed with 1 litre of nitric acid of 1.19 specific gravity, allowed to stand for twenty-four hours at about 35° C., and filtered.

(b) One hundred and fifty grammes of ammonium molybdate are dissolved in water in a litre flask, 400 grammes ammonium nitrate added, the solution made up to the mark with water, mixed with 1 litre of nitric acid of 1.19 specific gravity, allowed to stand for twenty-hours at about 35° C., and filtered.

(4) *Magnesia Mixture*.—One hundred and ten grammes of pure crystallized magnesium chloride and 140 grammes of ammonium chloride are dissolved in 1,300 c.c. of water, with the addition of 700 c.c. of 8 per cent. ammonia. The solution is allowed to stand for several days before filtering.

II. *Directions for Working*.—Five grammes of Thomas phosphate powder (in the condition as received) are placed in a half-litre flask, which is then filled up to the mark with diluted ammonium citrate solution at 17.5° C. The flask is closed with an indiarubber stopper, and at once placed in a rotating machine making thirty to forty revolutions per minute. The solution is kept agitated by the machine for thirty minutes, and is then promptly filtered off. Fifty c.c. of the filtrate are introduced into a beaker, together with 100 c.c. of molybdenum solution; the beaker is placed in a water-bath heated to 80° to 95° C., allowed to remain in the bath from ten to fifteen minutes, taken out, the solution cooled to the temperature of the room, and filtered. The yellow precipitate is then washed with a 1 per cent. nitric

acid solution, and dissolved in about 100 c.c. of a 2 per cent. ammonia solution (cold). The phosphoric acid is precipitated in this by 15 c.c. magnesia mixture added drop by drop with constant stirring. After standing for about two hours, the ammonium magnesium phosphate is collected upon a filter (unless the Gooch crucible is used), washed with a 2 per cent. ammonia solution, dried and ignited. For igniting the precipitate, a Bunsen burner is used until the filter is completely consumed (thirty to forty minutes), and afterwards a Rössler gas crucible furnace (for about two minutes).

III. *Remarks upon the above Method.*—(1) It is self-evident that the citrate solution must contain as nearly as possible the prescribed amount of citric acid and ammoniacal nitrogen. Deviations from the proper quantity have considerable effect upon the results.

(2) The citrate solution should be as nearly as possible of the ordinary mean temperature of the laboratory (17.5° C.). Fluctuations in temperature occasion serious mistakes. If the rotating machine be in a separate room, its temperature should be the same as that of the laboratory, so that the citrate solution shall undergo no change of temperature during the half-hour it is in the machine.

(3) The use of shaking machines instead of rotating machines has been proved to be inadmissible. Being of varied construction, they have not all the same power, and analyses made by the use of different machines have varied considerably. The machine made by the firm of Ehrhardt and Metzger of Darmstadt should be used. It is constructed of metal and driven by a hot-air motor.

(4) The machine should make not less than thirty and not more than forty revolutions per minute, and care should be taken that the half-hour is accurately timed. It is also important that the filtration should be commenced directly the vessel is taken from the rotating machine, as, if allowed to stand for any time before filtering, deviations either way in the results may take place.

(5) The filtration should be conducted as quickly as possible. Should the filtrate be at all turbid, it should be passed through the filter again, until quite clear. Too frequent filtration, however, should be avoided on account of prolonging the time.

(6) The phosphoric acid must be determined by the molybdate method; direct precipitation by magnesia gives high results when the percentage of silicic acid rises above a certain limit.

(7) No other molybdenum solution but the one described (containing ammonium nitrate) is to be used for precipitating the phosphoric acid.

(8) If the beaker is plunged deep into the water-bath, the phosphoric acid is completely precipitated in five minutes, though it is better to allow ten to fifteen. If, however, the digestion be continued as long as fifty minutes, incorrect results are obtained, in consequence of the precipitate becoming too much contaminated with silicic acid.

(9) The yellow precipitate should dissolve quickly and completely in the 2 per cent. ammonia solution, without warming. If the solution only becomes clear after long standing, it should be rejected, and the determination be recommenced in a fresh sample.

c. *Determination of the Percentage of Fine Meal.*—Fifty grammes of Thomas

powder are to be shaken for fifteen minutes in a No. 100 sieve, to be obtained from Amandus Kahl, Hamburg. The shaking may be done by hand or by means of a machine constructed for the purpose.

IV. *Determination of Potash in Potash Salts.**—A. *Analysis of Concentrated Potash Salts.*—(1) *Muriate of Potash.*—(a) *Determination of the Potassium Chloride.*—An aqueous solution is made by dissolving 7.6405 grammes of the finely prepared sample in 500 c.c. With salts containing more than 0.5 per cent. SO_3 , it is necessary first to convert the sulphates into chlorides by precipitation with solution of barium chloride, acidified with hydrochloric acid. Twenty c.c. of the solution = 0.3056 gramme of the salt, are placed in a flat porcelain basin of about 10 c.m. diameter, 5 c.c. of platinic chloride added, and the solution evaporated on the water-bath, until, when allowed to cool, the syrupy fluid quickly solidifies in fine crystals. The basin should be lifted off the bath frequently during the evaporation, and the fluid mixed by imparting to it a circular motion. The dry residue is then rubbed up quite fine with a glass rod, about 20 c.c. alcohol added, again stirred and filtered through a weighed filter. The filter should be previously washed with alcohol, and dried at 120° to 130° C., and it should be weighed warm. In filtering, care should be taken that the liquid first poured on does not touch the edge of the filter. The filtration can be expedited by the aid of a moderately powerful aspirating apparatus, and the double salt can be easily washed quite clean on the filter itself. The filter with the precipitate, after removal of as much as possible of the alcohol, by suction and pressing between blotting-paper, is dried at 120° to 130° C. until it ceases to lose weight. It is to be weighed warm. The drying occupies, as a rule, about twenty minutes. 0.001 gramme of potassium platinum chloride corresponds to 0.1 per cent. KCl.

(b) *Determination of the Sodium Chloride.*—12.5 grammes of the sample are dissolved in 25 c.c. of water, by boiling in a quarter-litre flask, after the addition of a little potassium carbonate to convert the magnesium and calcium compounds into carbonates, and the solution is then made up to the mark with absolute alcohol. It is now filtered, and 100 c.c. of the filtrate = 5 grammes substance, introduced into a platinum or porcelain basin, a few drops of concentrated hydrochloric acid added to convert any potassium carbonate into chloride, and the liquid evaporated to dryness; the residue is then gently ignited and weighed. In this mixture of potassium chloride and sodium chloride, either the potassium chloride is determined by means of platinic chloride in the usual way, and the sodium chloride taken by difference, or the mixed chlorides are titrated with a $\frac{N}{10}$ silver solution.

(c) *Determination of the Magnesium Chloride.*—Twenty-five grammes are dissolved in water in a half-litre flask, 10 c.c. normal potash solution added, and the flask made up to the mark. The liquid is then filtered, and 50 c.c. of the filtrate titrated with a $\frac{N}{10}$ sulphuric acid solution. Calcium compounds remaining in the solution have no influence upon the result.

(2) *Determination of Potassium Sulphate in Sulphate of Potash or Sulphate of Potash and Magnesia.*—8.9235 grammes of the finely prepared sample are dissolved in about 350 c.c. of water, with the addition of 25 c.c. concentrated hydrochloric

* Methods used in the Stassfurt potash works, and communicated by the syndicate.

acid by boiling in a half-litre flask. The sulphuric acid is then precipitated by the addition of barium chloride drop by drop to the boiling solution from a burette, its complete precipitation being indicated by a crystal of barium chloride producing no turbidity in the clear liquid, after the precipitate has settled out. Any excess of barium chloride must be got rid of by the addition of sulphuric acid. The solution is cooled, made up to the mark, and filtered, and to 20 c.c. of the filtrate = 0.357 gramme of substance, 5 c.c. of platinic chloride are added, and the determination continued in the usual way. One milligram K_2PtCl_6 corresponds to 0.1 per cent. K_2SO_4 . In testing samples of sulphate of potash, 0.3 per cent. is to be added to the percentage of potassium sulphate found, but with sulphate of potash and magnesia no correction is required.

(3) *Determination of Potassium Chloride or Sulphate in Calcined Potash Salts used as Fertilizers.*—15.281 grammes (for KCl) or 17.847 grammes (for K_2SO_4) are dissolved in water in a half-litre flask, with the addition of 10 c.c. concentrated hydrochloric acid, the solution made up to the mark, and filtered. Two hundred and fifty c.c. of the filtrate (corresponding to 7.6405 grammes or 8.9235 grammes, as the case may be) are transferred to a half-litre flask and further treated, by addition of barium chloride, as directed for the determination of potassium sulphate.

B. *Determination of Magnesium Sulphate in Kieserite.*—Ten grammes of the finely prepared sample are placed in a half-litre flask about two-thirds filled with water, and boiled for not less than an hour. The solution is then cooled, 50 to 60 c.c. of potash solution of double normal strength and 26 c.c. of a solution of neutral potassium oxalate (1 in 10) added, made up to the mark, allowed to stand a quarter of an hour, and filtered. Five hundred c.c. of the filtrate are then titrated back with a $\frac{N}{10}$ sulphuric acid solution. To the percentage of magnesium sulphate found 0.2 per cent. is to be added.

C. *Methods for the Examination of Mineral Potash Salts (Carnallite, Kainite, Sylvinite, and Mountain-Kieserite).*—(1) *Preparation of the Sample.*—In order to avoid differences arising through imperfect grinding, a large sample—if possible, at least half a kilo in weight—should be ground in a mill or mortar.

(2) *Determination of Potash by the Precipitation Method.*—35.70 grammes of kainite or sylvinite, or 30.56 grammes of carnallite or mountain-kieserite, are dissolved in 350 c.c. of water, with the addition of 10 c.c. of hydrochloric acid, by boiling in a half-litre flask. The solution is cooled, made up to the mark, and 50 c.c. precipitated with barium chloride in a 200 c.c. flask. Twenty c.c. of the filtrate (corresponding to 0.3570 gramme or 0.3056 gramme, as the case may be) are then evaporated with 5 c.c. of platinic chloride solution, and further treated in the usual way.

(3) *Complete Analysis of Mineral Potash Salts.*—One hundred grammes are dissolved by boiling in about 500 c.c. of water, filtered, the residue washed, and the filtrate and washings made up to 1 litre. A portion of this solution is used for making a gravimetric determination of sulphuric acid by precipitation with barium chloride, and another portion for the determination of the lime and magnesia. The alkaline chlorides are determined as follows: One hundred c.c. of the above solution, corresponding to 10 grammes of substance, are acidified with hydrochloric acid,

raised to the boil, the sulphuric acid precipitated by barium chloride (taking care not to use an excess), and the filtrate made up to a half-litre. Fifty c.c. of this, corresponding to 1 gramme of substance, are evaporated to dryness to expel hydrochloric acid, and the magnesium chlorides separated by ignition with oxalic acid. The residue, after ignition, must be moistened with a little ammonium carbonate to convert the caustic lime into carbonate. The alkaline chlorides, being completely freed from lime and magnesia, are now weighed, and then the potassium chloride determined in them by means of platinic chloride, using 10 c.c. The sodium chloride is taken by difference. In the case of kainite and sylvinit, the results are put together in the following manner: From the total soluble sulphuric acid found, that present as calcium sulphate is deducted; the soluble sulphuric acid then remaining is combined with the potash and magnesia in such a way as to form the two sulphates in molecular proportions, these salts being associated in this way in kainite and schönite; any remaining potassium or magnesium is then reckoned as chloride. In this way, the potassium present as kainite ($K_2SO_4, MgSO_4, MgCl_2 + 6H_2O$) or schönite ($K_2SO_4, MgSO_4 + 6H_2O$), and that present as chloride, are separately shown. The sodium is stated as chloride. In the analysis of carnallite and mountain-kieserite, the results are put together as follows: The lime is first combined with its proper proportion of sulphuric acid to form calcium sulphate, and the remainder of the sulphuric acid is then combined with magnesia; lastly, the magnesium not combined with sulphuric acid is reckoned as magnesium chloride.

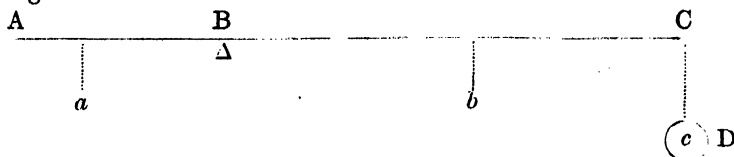
(To be continued.)

NOTE ON THE USE OF THE WESTPHAL BALANCE.

By A. MCGILL.

In using the Westphal balance on cylinder oils, I have had occasion to note the much greater sensitiveness of a particular instrument made by Westphal than of another made by Oertling. The former has a plummet weighing 4.3 grammes, and displacing 1.02 grammes water at $15^\circ C.$; the latter a plummet weighing 15.0 grammes, and displacing 6.5 grammes water. The specific gravities of these plummets are, therefore, approximately 4 and 2 respectively.

In attempting to explain the undoubted fact of greater sensitiveness in the first of these, I argue as follows:



If AC represents the beam of a Westphal balance with fulcrum at B and plummet at D,

let a = weight of short arm, acting at its centre of gravity.

„ b = „ longer arm, with suspended weights, acting at the centre of gravity of the system.

„ c = „ plummet in air.

„ c' = „ volume of liquid displaced by D.

The condition of equilibrium is :

$$a = b + (c - c') ;$$

and in order that motion may take place downwards at the plummet end, an additional weight (w) must be given, the magnitude of which will diminish with the viscosity of the liquid in which D is immersed, and inversely with the delicacy of the adjustments, friction of knife-edges, etc.

While the system is a rigid one—*i.e.*, so long as the wire suspending the plummet is tense—motion is produced by a force whose measure is the difference between a and $b + (c - c') + w$. But once this motion begins, the system may separate into two parts (owing to the flexibility of the wire suspending D, and to inertia); namely, into that part which moves in air, and the remainder of the system moving in the liquid in which D is immersed. The first part will have a tendency to move in a reversed direction under a force whose magnitude is $a - (b + w)$, while the plummet sinks under a force of magnitude $(c - c')$; whose value changes with the density of the plummet and that of the liquid, as well as with the absolute weight of the former.

I am convinced that the best results will be obtained by employing plummets of different weight and density, according to the different specific characters (viscosity, etc.) of the liquids operated on, and am well assured of the fact that for viscous oils the value of the ratio $c : c'$ should approximate 4 rather than 2. I have no doubt that the absolute value of c should bear some relation to w , and probably also to the density and viscosity of the liquid worked on. So far as this relation to w is concerned, it will evidently vary with each balance; but the relation to the liquid might be determined experimentally once for all.

LABORATORY OF THE INLAND REVENUE DEPARTMENT,
Ottawa, March 31, 1896.

NOTE ON HEHNER'S TEST FOR FORMIC ALDEHYDE.

BY NORMAN LEONARD, B.Sc., F.I.C.

IN trying Hehner's test for formic aldehyde in milk, which consists in the production of a blue or violet colour when formalized milk is poured on to the surface of strong sulphuric acid, it was found that the reaction could be easily obtained with commercial sulphuric acid, but quite failed when the pure redistilled acid was used.

In order to identify the impurity present in commercial sulphuric acid, to which Hehner's reaction is due, the effect of adding common impurities to the redistilled acid was ascertained. Nitric acid, nitrous acid, hydrochloric acid, arsenious acid, and lead sulphate were found to have no effect, but the addition of a trace of ferric chloride at once conferred upon the pure acid the power of giving the violet colour with formalized milk. Platinic chloride answered equally well, while mercuric chloride, bromine, and potassium permanganate were less effectual. No reaction could be obtained by the use of sodium hypochlorite, of ferrous sulphate, or of potassium bichromate. The commercial acid was found to give distinct reactions with potassium ferrocyanide and with ammonium sulphocyanide, whereas no trace of iron could be detected in the pure redistilled acid.

It would therefore appear that the presence of a feeble oxidizing agent, such as a metallic perchloride, is necessary for the production of Hehner's reaction. The test is not improved by the addition of any considerable amount of ferric chloride, but rather the reverse. A trace only is necessary and sufficient.

NOTE.—Mr. Hehner informs us that he used "pure" or "redistilled" sulphuric acid in all his tests, but he now finds that a trace of ferric chloride renders the reaction far more distinct.—EDITOR.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD ANALYSIS.

Estimation of the Volatile Acids in Wine. L. Magnier de la Source. (*Ann. de Chimie Analyt.*, i., 65-68.)—The following modification of the Muller method is recommended by the author, as giving very exact results with less trouble and attention than are required in the original process. It is also quicker than the author's evaporation *in vacuo* process, proposed as a modification of the Muller method.

To compensate for the somewhat slower rate of evaporation in air compared with that in a vacuum, the exposed surface of the liquid is increased. To this end two evaporating dishes of about 4 centimetres radius and 2 centimetres in depth are employed, in each of which 5 c.c. of the wine, previously freed from carbonic acid, is placed. One of them is then put into a desiccator containing both concentrated sulphuric acid and caustic potash, and the other, after the addition of five drops of alcoholic phenol-phthalein solution (0.5 gramme per 100 c.c.), is neutralized with baryta water, 1 c.c. of which corresponds exactly to 0.004 gramme of sulphuric acid. The colour of the wine turns directly, and as soon as it passes into violet the reaction is complete, the number of c.c. of baryta used indicating the total acidity in terms of sulphuric acid.

The wine in the desiccator is left to evaporate for two days at a temperature of about 15° C., by which time the extract will have become practically dry. It is then taken up with 2 c.c. of warm water, and left to re-evaporate for another two days. The acidity being determined as in the other case, the difference in the amount of baryta used expresses (as sulphuric acid) the acidity due to the volatile acids. The volatilization of the acetic acid is so complete, that a wine partially transformed into vinegar, and having a total acidity of 29.80 grammes per litre (as H_2SO_4), left behind after this treatment only 1.60 grammes of fixed acid.

The method is also applicable, with a certain amount of modification, to wines whose acidity has been partly masked by the addition of an alkali or a neutral tartrate. In such cases, instead of operating direct on the wine, about 2 c.c. of a freshly-prepared solution of tartaric acid, containing some 25 grammes per litre, are first added to each basin, care being taken that the two volumes of added acid are exactly equal. This allows the displacement of the volatile acids, and their amount can be determined by difference as before. This will always represent the *original* acidity of the wine before it was doctored.

Whether the wine has been neutralized or not, the proportion of volatile acids will come out higher under this last mode of treatment—an effect that may be due to several causes, such as partial displacement of the acid radical in the potassium chloride by the added tartaric acid, or by a disturbing of the equilibrium which exists between the last traces of acetic acid and the natural tartrates in the wine; but in all cases where neutralization has been effected the results will be so decided as to be unmistakable, as is shown by the following examples :

NATURAL WINES.

		Degree of Acidity due to Volatile Acids.	
		By Direct Determination in the Wine.	Determined after the addition of Tartaric Acid in Excess.
Gironde wine...	...	0.52 gramme per litre	... 0.67
Sour	2.56 " "	... 2.64
"	2.32 " "	... 2.60
Tunis wine, unsound	...	2.58	... 2.88
Algerian wine	...	1.56	... 1.76
" "	...	1.47	... 1.72

WINES PARTLY NEUTRALIZED.

By Seignette salt ...	0.68	" "	... 1.40
By neutral potassium tartrate ...	2.40	" "	... 3.33
By ammonia...	2.0	" "	... 2.72

C. S.

Notes on Lard, etc. C. Fresenius. (*Chem. Zeit.*, 1896, xx., 129.)—The present author agrees with Goske that the iodine test is not of much use for detecting adulteration in lard when the added oils and fats are of fair purity. Becchi's test is fairly reliable, and the wine-colour that often appears must not be ignored; for even after a thorough washing in dilute acetic acid, a lard containing cotton-oil gives some reaction, and only in the presence of very small quantities of oil does the liquid remain colourless. An examination of the crystalline form is strongly to be recommended. Mixed fats should not be tested as such for their melting and solidification points; but the fatty acids should be prepared.

The author prefers the following process: The sample is introduced into a test-tube 2.5 c.m. wide and 10 c.m. long placed in a beaker filled with cotton-wool. The temperature of the laboratory should be 17 to 20° C., the fat heated to 40° C. and allowed to cool at the rate of 1° per ten minutes. The point to which the thermometer first falls is called the "critical point." The time during which it remains constant is noted, and also the point to which it again rises. If more than 15 per cent. of oil alone is added to lard, no period of constant temperature is noticed; while in the case of adulterated samples, if the critical point is above 26° C., tallow, with or without oil, has been added; and if below 24° C., oil only has been employed. The annexed table shows the results obtained on a number of products, the "period of rest" indicating the time in minutes of constant temperature as shown by the thermometer in the fat:

			Critical point.	Period of rest.	First rise to.	Period of rest.	Second fall to.	Period of rest.	Second rise to.	Period of rest.
Butcher's lard	27.6	2	29.4	2	—	—	—	—
"	27.4	2	28.3	10	—	—	—	—
"	26.6	2	29.3	2	—	—	—	—
American steam lard	25.5	3	26.3	3	—	—	—	—
"	"	...	25.2	2	25.9	3	—	—	—	—
"	"	...	24.6	3	25.5	3	—	—	—	—
"	"	...	24.4	2	24.9	3	—	—	—	—
95% lard, 5% oil	23.9	2	24.1	3	—	—	—	—
90	"	10	23.3	3	23.7	2	—	—	—	—
90	"	10	31.1	3	—	—	23.3	8	—	—
90	"	8	24.0	7	—	—	—	—	—	—
85	"	10	28.3	5	—	—	22.9	9	—	—
82	"	10	29.6	2	—	—	22.5	8	—	—
80	"	10	31.7	4	31.9	3	22.9	7	23.3	15
77.5	"	15	30.6	2	31.0	2	22.7	5	23.2	20
70	"	20	30.3	4	30.5	2	21.9	6	22.1	6
62.5	"	25	30.0	4	30.5	2	21.6	2	21.8	5
67	oil,	33	38.0	10	38.9	3	—	—	—	—
60	"	40	31.0	10	—	—	—	—	—	—
10	lard,	90	36.5	5	37.4	5	—	—	—	—
85	"	10	—	—	—	—	—	—	—	—
	5	tallow, etc.	26.0	10	—	—	—	—	—	—

F. H. L.

TOXICOLOGICAL ANALYSIS.

Detection of Hydrocyanic Acid in Cases of Poisoning. F. Filsinger. (*Chem. Zeit.*, 1896, xx., 305.)—A case of supposed poisoning by potassium cyanide has recently occurred in Dresden, in which it was necessary to examine the stomach, etc., of the corpse ten days after death. After proving the absence of inert cyanides, ferro- and ferri-cyanides by means of the iron and copper tests, the different articles were distilled, according to Dragendorff's process, on the water-bath in presence of tartaric acid. The distillates were examined for the formation of Prussian blue, ferric thiocyanate, and by Schönbein's reaction. In the stomach and its contents hydrocyanic acid was readily detected by all three tests; in fact, it would have been possible to conduct the operation quantitatively. The smaller intestines and the blood reacted strongly with Schönbein's test, giving a powerful blue colour, but the other reactions were uncertain. A small brownish, alkaline deposit found in a tumbler that had evidently contained the wine in which the potassium cyanide was dissolved also gave Schönbein's test very plainly; so that this process, in spite of recent statements to the contrary, seems to be the best to adopt in similar cases.

F. H. L.

ORGANIC ANALYSIS.

The Oil of the Egg. P. Paladino and D. Toso. (*Gior. di Pharm. di Chem.*; through *Jour. de Pharm. and Chim.*, 1896, 247-249.)—This oil, which is used in ointments, is extracted from the dried yellow of the egg either by pressure or by solvents. The normal composition of eggs is 60 per cent. of white and 40 per cent. of yolk, the latter containing from 25 to 35 per cent. of oil. In preparing it the authors separated the yolks (900 grammes) of sixty-eight hard-boiled eggs, heated them rapidly in a porcelain dish, and extracted the oil by pressure.

As thus obtained the oil was perfectly limpid and of a yellow colour. On cooling, it became viscous, and deposited a crystalline sediment. It readily became rancid and lost its colour on exposure to air and light.

Its physical constants were:

Density	0.9156 at 20° C.
Solidifying-point	8-10° C.
Melting-point	22-22.5° C.
Fatty acids (melting-point)	34.5-35° C.
Saponification number	185.2 to 186.7.
Iodine number	81.21 to 81.6.

The melting was often incomplete, owing to the presence of crystals melting at 145°, which probably consisted of cholesterin.

C. A. M.

Technical Analysis of Asphaltum. No. 2. Laura A. Linton. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 275-283.)—In this paper Miss Linton gives an account of further work on this subject (ANALYST, xx., 41), which has led her to modify her methods in some respects. She finds that the old method of determining moisture does not give correct results, since not only water but also portions of petrolene volatile below 100° C. are expelled. This may be obviated by drying at 50° C., experiments proving that all the moisture may be driven off at that temperature. At the same time it is urged that, since the moisture is of hygroscopic origin and varies with the atmospheric conditions, it should never be estimated as a constituent part of the asphaltum. To obtain comparative results it is advisable to air-dry all samples to constant weight, and thus entirely exclude water from their percentage composition.

A mechanical improvement consists in the use of weighed filter-paper instead of an Erlenmeyer flask, the digestions being carried out in separating funnels. The only precaution necessary is to see that the solution in the funnel is not too concentrated before running from the tap, since this causes precipitation of petrolene. This is readily obviated by frequently changing the petroleum spirit at first, until the greater part of the petrolene is removed, when it is safe to leave it for longer periods.

By fractionation of the asphaltene into the portion soluble in boiling turpentine and that soluble only in chloroform, Miss Linton considers that figures are obtained which probably indicate the relative altering or "ageing" of different varieties of asphaltum. The process, which is very tedious, often taking from one to four weeks,

is as follows: After complete removal of petrolene, the filter is digested with boiling turpentine until the filtrate is colourless, the contents of the filter being then washed in alcohol and dried at 100° C. The dried residue should be a loose brown powder without coherence. If otherwise, the extraction with turpentine must be repeated possibly several times. The part of the asphaltene soluble in boiling turpentine is a black viscid semi-liquid substance resembling tar and melting at or below 100° C.

The percentage results in the following table show that not only do the "aged" varieties of asphaltum contain a larger percentage of asphaltene, but that the turpentine fraction becomes a smaller proportion of the total bitumen, while the chloroform fraction becomes larger:

Variety.	Petrolene.	Turpen- tine Fraction.	Chloro- form Fraction.	Asphal- tene.	Total Bitumen.	Ratio CHCl ₃ Fraction to total Bitumen.	Organic Matter not Bitumen.	Mineral Matter.
1. Average land asphaltum, Trinidad ...	33.73	15.67	3.179	18.849	52.579	1 : 16	11.528	35.886
2. Altered or "aged" (iron pitch) land, Trinidad	33.574	13.7	9.627	23.327	56.901	1 : 6	8.414	34.684
3. "Aged," land, "	21.362	15.2	15.112	30.312	51.674	1 : 3	9.85	38.375
4. Average, lake, "	35.40	12.30	5.287	17.587	52.987	1 : 10	10.962	36.100
5. "Aged," lake, "	26.925	18.613	6.687	25.3	52.225	1 : 8	11.237	36.537
6. " " " "	19.25	22.35	10.962	33.312	52.562	1 : 5	9.562	37.875
7. "Aged" (iron pitch), lake, Trinidad ...	22.25	9.785	12.54	22.325	44.575	1 : 4	8.937	46.462
8. Asphaltum, Texas ...	7.538	1.601	trace	1.601	9.139	—	—	90.861
9. Turrellite, "	8.786	3.267	trace	3.267	12.053	—	—	87.947
10. Asphaltum, Indian Territory ...	9.503	0.990	trace	0.990	10.493	—	—	89.506
11. Graphamite, W. Va. ...	49.959	17.458	32.583	50.041	100.00	1 : 3	—	—
12. Scyssel asphaltic rock, France ...	7.486	3.945	0.371	4.316	11.802	1 : 31	—	88.198

C. A. M.

Estimation of Sulphur in Petroleum. R. Kissling. (*Chem. Zeit.*, 1896, xx., 199).—This investigation was carried out without knowledge of Heusler's work on the same subject (*ANALYST*, xx., 187), and is essentially but a modification of his process. The petroleum was burnt in a lamp fitted with a chimney contracted and bent to an acute angle at the top, to which were connected a 200 millimetre U-tube filled with glass beads, and also two of Will and Varrentrapp's nitrogen bulbs. A 5 per cent. solution of permanganate was employed to absorb the sulphur, preliminary experiments having shown that a mixture of sodium carbonate and potassium nitrate was not efficient. The beads were moistened with 15 c.c. of the liquid, and 5 c.c. were placed in the bulbs. The lamp was so arranged that a current of 300 c.c. of air drawn through the apparatus per minute was sufficient to maintain perfect combustion. After boiling the resulting solution with HCl, it was found necessary to

cool it well before filtration, to remove hydrocarbons; and if much lamp-black had formed, it proved advisable to repeat the test, although the liquid could be evaporated to dryness, and ignited in presence of an oxidizing substance. The experiments lasted from eighteen to forty-six hours, and the oil burnt varied from 26 to 107 grammes. No special provision was made to remove sulphur compounds from the air admitted to the lamp, but care was taken to prevent alterations in the purity of the atmosphere. Some twenty-five samples were examined, and the results are summarized in the following table, the figures being the weight in grammes of the sulphur in 1,000 grammes of the oil burnt:

Description of Sample.					Sulphur. Per 1,000.
"Lima" petroleum	0.448
Pennsylvanian petroleum	0.278
" (from another works)	0.271
"Kaiseroel"	0.105
"	0.098
"Safety oil" (85° Abel), specially refined	0.193
Alsatian oil	0.589
Baku oil	0.297
Galician oil	0.388
" (from another works)	0.233
"Kronenoel" from Pennsylvania, specially refined	0.190

F. H. L.

Molybdic Acid as a Test for Alcohol. E. Merck. (*Chem. Zeit.*, 1896, xx., 228.)—The author proposes the following modification of Davy's test: Pure molybdic acid is dissolved in warm strong sulphuric acid, and the resulting solution poured through the liquid under examination in a test-tube, both being kept as nearly as possible at a temperature of 60° C. In presence of alcohol, a distinct blue ring appears at the junction between the two liquids, which is the more intense the larger the proportion of alcohol present. On shaking, the colour disappears, but by addition of a further quantity of the reagent it may be reproduced. The test is, of course, not characteristic of alcohol only, but it will detect even 0.02 per cent. of ethyl alcohol, and 0.2 per cent. of methyl alcohol in aqueous solution.

F. H. L.

Estimation of Morphine in Opium. G. Loeff. (*Apoth. Zeit.*, 1896, ii., 192; through *Chem. Zeit. Rep.*, 1896, 114.)—Five grammes of the sample in fine powder are rubbed down with 5 c.c. of water, washed into a tared flask, and diluted to 44 grammes in (nett) weight. After shaking for fifteen minutes, 1 gramme of sodium salicylate is added, the whole shaken again and filtered. This treatment removes the substances which usually hinder the precipitation of the alkaloid. 25.8 grammes of the filtrate (= 3 grammes of opium) are then treated with 3 grammes of ether and 1 of ammonia. The mixture is agitated for ten minutes, the morphine collected on a small filter, the flask rinsed twice with 5 c.c. of water, and the precipitate washed therewith and dried. It is finally extracted with benzene, dried again, and weighed.

F. H. L.

Detection of Resin and Rosin Oil in Oils and Varnishes. F. Ulzer. (*Mittheil. techn. Gew. Mus. Vienna*, 1896, 91; through *Chem. Zeit. Rep.*, 1896, 146.)—A very weak solution of pure linseed-oil varnish in acetic anhydride gives a dark, reddish-brown colour on addition of sulphuric acid. In certain cases it is better to shake up the sample with absolute alcohol, and after several hours to remove and evaporate the spirit, treating this residue with acetic anhydride and sulphuric acid. The Storch-Morawski (*cf. ANALYST*, xvi., 155) process is very useful in the examination of varnishes, and it is capable of showing the presence of even comparatively small amounts of resin, the reaction being particularly suited for detecting the addition of cheap resins such as rosin to copal and amber varnish. F. H. L.

Estimation of Nitrogen in Guano. E. Franke. (*Chem. Zeit.*, 1896, xx., 325.)—Haselhoff has lately thrown some doubt on the Jodlbauer method of conducting this analysis, and has suggested that the sample should be extracted with water on a filter, the nitrogen existing as ammonia and nitrates being determined in the solution by Ulsch's process, and the organic nitrogen in the residue according to Kjeldahl. It is at once evident that this modification is inexact, for the guano contains easily soluble organic nitrogen compounds which are not completely converted into ammonia by the action of caustic soda. The author has examined several samples of guano by the Ulsch-Kjeldahl process applied to the manure itself, and also by that of Jodlbauer, and has compared the figures obtained with those of Haselhoff's modification, and finds the latter considerably too low.

Experiments on guano to which sodium nitrate had been added, showed that the Jodlbauer process was exact up to a certain limit (9 per cent.), but above that the phenol sulphonic acid treatment gave too low a yield: the Ulsch-Kjeldahl method was always perfectly satisfactory. The author concludes that as guanos seldom contain more than 2 per cent. of nitrates, the former is perfectly reliable, that Haselhoff's suggestion is useless, and that in all doubtful cases recourse should be had to reduction by means of iron and sulphuric acid (Ulsch), followed by the Kjeldahl process. F. H. L.

A New Test for Dulcin. A. Jorissen. (Reprint from the *J. Pharm. de Liège*; through *Chem. Zeit. Rep.*, 1896, 114.)—This substance is a derivative of saccharin, and is already being used to sweeten various beverages. The sample is suspended in water in a test-tube, and 2 or 3 drops of a solution of mercuric nitrate, containing no free acid, and preferably made from the freshly-precipitated oxide, added. The tube is plunged into boiling water for five or ten minutes, when a faint violet tint should appear. On introducing a small quantity of lead peroxide, a fine violet colour is momentarily produced. The reaction is capable of detecting 0.001 gramme of dulcin. F. H. L.

INORGANIC ANALYSIS.

An Electrolytic Method for the Determination of Mercury in Cinnabar. W. Rising and V. Lenher. (*Journ. Amer. Chem. Soc.*, xviii., 1896, pp. 96-98.)—To avoid the objections to the employment of aqua regia as a solvent for cinnabar, the author proposes the use of hydrobromic acid. This is made by treating potassium bromide with sulphuric acid of 56° Baumé (s.g. 1.364), and conducting the gas into water. As thus prepared the hydrobromic acid contains no free bromine. The solution of cinnabar is nearly neutralized with caustic potash, pure potassium cyanide added, and electrolysed in a weak current (0.025 amperes per square centimetre), when the mercury is deposited on a platinum dish used as a negative electrode. The results quoted are concordant, and agree well with those obtained with aqua regia as the solvent.

C. A. M.

Estimation of Mercury with Sodium Peroxide. M. C. Schuyten. (*Chem. Zeit.*, 1896, xx., 239.)—This process has been tried on both the chlorides, the sulphate, nitrate, and red oxide of mercury. It is also available for organic compounds, but cinnabar is not attacked. The substance is weighed out into a porcelain basin, covered with water, and an inverted funnel with a neck bent at right angles placed over the vessel. The peroxide is then added by degrees until no further precipitate is formed. The whole is warmed gently, cooled, and the metallic mercury filtered off. During drying, the desiccator must be kept in the dark at a low temperature, and the funnel should be covered with paper. Three tests on recrystallized mercuric chloride gave a mean of 73.52 per cent. of Hg (theory, 73.92 per cent.). If the filtrate is neutralized, the halogen it contains can be titrated direct, according to Volhard's method.

F. H. L.

Detection of Mercuric Cyanide. D. Vitali. (*Boll. Chim. Farm.*, 1896, xxxiv., 737; through *Chem. Zeit. Rep.*, 1896, 72.)—On warming a solution of mercuric cyanide with magnesium (ribbon), hydrogen is evolved, which soon begins to smell of hydrocyanic acid. If the action is only allowed to proceed for a short time, a mixture of mercury and mercurous oxide is produced; but if it is continued as long as any gas is set free, the mercury will be found only in the metallic state, while much magnesium cyanide can be detected in the liquid.

In cases of supposed poisoning, the material is mixed with magnesium powder, and introduced into a tubulated retort fitted with a safety-tube and a well-cooled condensing arrangement containing caustic soda. The retort is heated over a sand-bath as long as gas is given off; but before the mass becomes dry, acetic acid is gradually added to decompose the magnesium cyanide. The whole of the hydrocyanic acid distils over, and the mercury can easily be detected in the residue.

F. H. L.

Influence of Magnesia Mixture on Glass. L. L. de Koninck. (*Chem. Zeit.*, 1896, xx., 129.)—The author has repeated his earlier experiments on the action of magnesia mixture on glass, using a new Erlenmeyer flask of Jena glass 135 millimetres high, with a superficial area of 1,625 square centimetres. On allowing an old and perfectly clear solution to remain for thirteen months in the vessel, it was found considerably attacked, the magnesium silicate removed weighing, after ignition, 0.1525 gramme (=0.2168 of air-dried silicate), and corresponding to a loss of 1.5 milligrammes per square centimetre. With Stas's glass this was proved less than 1 milligramme per square centimetre in seventeen months.

In Ostwald's laboratory it is the custom to treat all glass apparatus that is to be employed for exact work with steam. This appears to alter the nature of the surface of the material so that it is no longer attacked by reagents; in fact, after fifteen months a flask so treated was practically untouched by the magnesia mixture. It should be observed that if the first layer of silicate be removed, and an ordinary flask submitted to the action of the liquid for a second time, it is attacked even more rapidly than before.

F. H. L.

The Quantitative Estimation of Arsenic in Crude Concentrated Sulphuric Acid. G. Hattensauer. (*Zeit. angew. Chem.*, 1896, 130-131.)—The usual method of making this estimation is to dilute the sulphuric acid, to filter off the precipitated lead sulphate, and to precipitate the arsenic in the filtrate. But this is tedious, and, according to the author, inaccurate for small amounts of arsenic.

The following method is based on the fact observed by Neher (*Zeit. anal. Chem.*, xxxii., 45) that arsenic is completely precipitated from strong hydrochloric acid solution by sulphuretted hydrogen: 500 c.c. of the strong acid (of specific gravity, say, 1.815 at 22° C.) are diluted with 500 c.c. of water, the mixture having a gravity of 1.46 at 15° C. After cooling, 500 c.c. of this are mixed with 500 c.c. of dilute hydrochloric acid (1 : 2), which is sufficient to prevent the precipitation of the lead as sulphate, and subsequently as sulphide. Sulphuretted hydrogen is then passed in for about one hour, the precipitate which is in the form of arsenic pentasulphide filtered off, and the arsenic determined as sulphide, or, after oxidation, as arsenic acid. The author found the precipitate quite free from lead, although in one case the amount of lead present in the acid was ten times that of the arsenic. The filter-paper is not attacked by the acid when diluted to the above-mentioned degree.

C. A. M.

Action of Ammonium Citrate on Basic Slag. O. Foerster. (*Chem. Zeit.*, 1896, xx., 131.)—The explanation of the abnormal behaviour of the three slags quoted by Gerlach and Passon (*ANALYST*, xxi., 81), in giving up more phosphoric acid to free citric acid than to ammonium citrate, is explained by the fact that the former is a much more energetic solvent of all phosphates than the latter. The difference is only likely to be observed in analysis when the samples examined contain difficultly soluble phosphates, such as the tricalcium salt or the compound $(\text{Ca}_3\text{P}_2\text{O}_8)_3\text{CaO}$, that yield up more phosphoric acid to free citric acid than to ammonium citrate. Most slags, however, do not contain these substances; their "unavailable" portion

consisting of iron or aluminium phosphates, which not only are much less soluble, but behave more similarly to both reagents.

F. H. L.

Estimation of Copper by the Cyanide Process. G. Denigés. (*Bull. Soc. Pharm., Bordeaux*, 1896, p. 18, through *Ann. de Chimie Analyt.*, i., 70, 71.)—The Parkes method may be improved by operating in an ammoniacal solution kept at boiling temperature throughout, the reaction being thereby rendered more regular and less dependent on the medium, without affecting the accuracy of the result.

Ten c.c. of the copper solution are placed in a porcelain capsule, along with an equal bulk of ammonia and made up to 40 c.c. with water. When the boiling-point is attained, a solution of cyanide of potassium, standardized on a one-tenth normal solution of argentic nitrate, is added quickly, drop by drop, until the blue tint has become very weak. Thenceforward an interval of three or four seconds should elapse between the drops until the decolorization is complete, care being taken that the ebullition is uninterrupted and brisk throughout. Doubling the volume of ammonia, or the presence of ammoniacal salts up to 1 or 1.5 gramme, does not affect the result.

One c.c. of the standardized cyanide of potassium corresponds to one ten-thousandth of a molecule of copper salt multiplied by 0.594 (the theoretical equivalent is 0.500). The metallic copper can be determined by the calculation

$$(n - 0.1) \times 0.594 \times 0.00635,$$

n being the c.c. of cyanide solution used, and 0.1 c.c. a constant arrived at by the author, as corresponding to the accuracy of the results.

C. S.

REVIEW.

PETROLEUM. By BOVERTON REDWOOD, assisted by G. T. HOLLOWAY. London: C. Griffin and Co., Limited. 2 vols. Price £2 2s.

THIS treatise, as stated in its sub-title, deals with "the geographical distribution and geological occurrence of petroleum and natural gas; the physical and chemical properties, production and refining of petroleum and ozokerite; the characters and uses, testing, transport and storage of petroleum products; and the legislative enactments relating thereto; together with a description of the shale-oil and allied industries."

A scheme so considerable needs the aid of careful classification for its execution, and such classification has been successfully carried out. The book is divided into sections of moderate size, each section being confined as closely as is practicable to a single distinct subject. Reference is thus facilitated, and the book made suitable for those needing speedy information on a specific point.

Vol. I. is chiefly concerned with historical and geographical matter, with details of methods of winning petroleum, and with accounts of manufacturing processes for the refining of the crude material.

In the second volume occurs that part of the work which is of more immediate interest to the readers of the ANALYST. Nearly 130 pages are occupied with a

description of the testing of petroleum and its products, and a large amount of information has been collected. The evaluation of crude petroleum is somewhat briefly discussed, the impression (a correct one, be it said) left on the reader's mind being that no standard method can be usefully laid down, and that the operator must use a modicum of brains. The section on methods and apparatus for determining the flashing-point of mineral oil is full of detail concerning the many forms in which the test is applied. The determination of viscosity is also treated of at length, many forms of apparatus, some well-devised and useful, others sufficiently obscure and likely to remain so, being described and freely illustrated. The section on mechanical tests of lubricating oils by the use of sundry frictional testing machines is similarly full, although the author concludes most justly that, save for special purposes, the utility of a frictional machine is small, and the method of choosing an oil by means of frictional results on a laboratory apparatus is altogether inferior to that of making selection by the aid of a knowledge of the viscosity of the lubricant. In the whole of the analytical part care has been taken to get together ample data; it is opinion rather than fact which is lacking. Of half a dozen apparatus or processes described, the expert knows well enough that only one or two are to be commended, but the non-specialist needs guidance. No one is better qualified than Mr. Redwood to state authoritatively what are, and what are not, useful methods, and this portion of the book would gain greatly in value were he to do so.

Such uses of petroleum as the production of "air-gas" and of oil-gas, and as fuel for prime-movers, including the modern autocar, are dealt with in the later portion of the volume, which concludes with a reprint of the regulations in use in civilized countries for the safe handling of petroleum, and with a statistical section.

The book, as a whole, is a storehouse of facts well classified and presented, but the reader must provide his own mental pepsin. B. B.

CORRESPONDENCE.

To the Editors of THE ANALYST.

DEAR SIR,—In the April number of THE ANALYST I note that in Messrs. Droop Richmond and Boseley's paper on "The Detection of Formalin" the authors have misquoted my figures published in THE ANALYST, xx., p. 157, as to the quantity of formalin present in milk samples. There are, however, two misprints in my paper which I should like to correct. I state correctly that "5 ounces of formalin to 1 gallon, corresponding to 2 ounces formaldehyde in 160 ounces, is used for making the milk formalin solution"; but the alternative, "or 1 : 320," should read, "1 : 80." The second misprint is " $\frac{1}{2}$ pint," which should read "1 gill or 5 ounces," and I find that in my original MSS. the words "one gill"* were altered to " $\frac{1}{2}$ pint" by yourself. The calculation deduced from the above data is correct, viz., 1 part of formaldehyde in 46,080 parts milk, assuming that a churn is 18 gallons as a maximum.

Yours faithfully,

SAMUEL RIDEAL.

28, VICTORIA MANSIONS, WESTMINSTER, S.W.

* A gill is a somewhat indefinite measure, since it means half a pint in some parts of the country.—EDITOR.

THE ANALYST.

JULY, 1896.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday, June 3, in the Chemical Society's Rooms, Burlington House, the President (Dr. Stevenson) occupying the chair. The minutes of the previous meeting were read and confirmed.

Mr. Hudson E. Kearley, M.P., was elected an honorary member. The following gentlemen were proposed for election as members of the Society: Frederick James Montague Page, B.Sc. (Lond.), Assoc. R.S.M., F.I.C., Lecturer on Chemistry at London Hospital; and Charles George Matthews, F.I.C., Analyst and Consulting Chemist, 58, Charing Cross, London.

The following papers were read: "The Analysis of Mortar," by W. J. Dibden and R. Grimwood; "Commercial Cream of Tartar," by Alfred H. Allen; "Note on a new form of Cheese Adulteration," by F. W. Stoddart; "Commercial Prussian Blue," by Ernest J. Parry, B.Sc., and John Henry Coste.

NOTE ON THE COMPOSITION OF TWO DEPOSITS FORMED IN WATER-PIPES.

By J. A. AND E. W. VOELCKER.

(Read at the Meeting, May 6, 1896.)

As in considering the suitability of any water for general domestic use, the question of its action upon metallic pipes is one of great importance, we venture to bring before the Members of the Society two cases which may be of interest.

The first case to which we would refer is one that occurred recently, and which shows how the action of a soft water on galvanized iron pipes may give rise to serious inconvenience.

We had lately occasion to examine a deposit which had formed in a pipe connected with the hot-water apparatus of a private house in the country. The pipe in question was completely blocked up with a white deposit, and the owner of the house was very disappointed to find that the water which we had previously reported to be a very soft and pure one, and suitable for general domestic use had, after a comparatively short time, given rise to a deposit in the hot-water pipes.

The owner was under the impression that the deposit was due to lime. Upon analysing the material we found that it contained no lime, but consisted almost entirely of a basic carbonate of zinc, together with oxide of zinc.

The analysis of the deposit was as follows :

Oxide of iron ...	0.56
Alumina ...	0.59
Basic carbonate of zinc ...	64.32
Oxide of zinc ...	21.96
Silica ...	4.07

On inquiry we found that the water was lifted by a ram to the height of 300 feet, along about half a mile of galvanized iron pipes. These appear to have been very rapidly attacked by the water, the zinc being dissolved and deposited in the pipes at those points where the water was very hot, and also in the boiler and hot-water cylinder.

The next instance shows the action upon iron of a very hard water containing a good deal of sulphate of lime and organic matter.

The well-water in question contained over 40 grains per gallon of total solids.

It was noticed that on the pump-rods in the well a black scale was formed, and in the stand-pipe there was a considerable amount of deposit.

The analysis of the scale and of the deposit revealed the presence of a notable quantity of free sulphur.

In the deposit in the stand-pipe the free sulphur amounted to over 5 per cent. This, probably, is to be accounted for by the action of the organic matter on the sulphates in the water, sulphuretted hydrogen being produced and then absorbed by the hydrated oxide of iron formed by the rusting of the iron pipes, while the subsequent oxidation resulted in the liberation of sulphur, the action being the same as takes place when the oxide from gas-purifiers is exposed to the air.

The analyses were :

	Deposit in Stand-pipe.	Scale on Pump.
Ferrous oxide ...	11.31	4.78
Ferric oxide ...	38.09	
Ferrous sulphide ...	2.72	2.64
Lime ...	7.11	48.63
Carbonic acid ...	11.45	37.08
Sulphuric acid ...	1.42	0.46
Free sulphur ...	5.20	1.65
Silica ...	8.55	0.90
Alumina ...	2.31	0.93

DISCUSSION.

The PRESIDENT said that the explanation given of the presence of free sulphur was very interesting, though at first sight it might not appear to be quite satisfactory. Of the actual presence of free sulphur, however, there could be no doubt. A good deal of interest attached to the deposit in the pipe, on account of the very highly basic character of the carbonate. Apparently it was not the ordinary basic carbonate which was usually formed on the surface of galvanized pipes and on zinc; that was a much less basic substance than this. Roughly speaking, the ratio of zinc to CO_2 was about $4\frac{1}{2} : 1$, which was quite exceptional, and seemed to point rather to a mixture of zinc oxide and the ordinary basic carbonate of zinc than to any other very definite chemical compound.

Dr. J. A. VOELCKER said that upon first inspecting the pipe in question, he naturally thought that the deposit was of the ordinary nature, viz., carbonate of lime. His brother (Mr. E. W. Voelcker), however, noticed that it was particularly soft; this led to a further examination of the deposit, when it was found that there was no lime at all in it, but that it consisted mainly of carbonate of zinc.

On referring back to the analysis of the water, which had been made some time previously, it was found that the water was distinctly soft, if containing only about 6 grains of total solids per gallon.

The portions of the pipes upon which the deposit had mostly formed were those nearest the boiler. The deposit contained something like 64 per cent. of basic carbonate, and some excess of oxide of zinc, which might have been due to change in the basic carbonate consequent on the heating.

The other water, of course, was of a different kind. The instance seemed to be of interest, as showing clearly the production of sulphuretted hydrogen in waters containing a good deal of organic matter, together with calcic and other sulphates.

Dr. DYER inquired whether the acidity of the first water had been taken with phenolphthalein. It might be interesting to do this.

Mr. E. W. VOELCKER said that the acidity had not been determined.* The water was from a Welsh mountain-stream, and contained very little organic matter. The oxygen absorbed was a mere trifle, and the water was not coloured like a peaty water would be.

Mr. KITTO inquired what amount of chlorine was present.

Mr. E. W. VOELCKER said the chlorine amounted to about 1 grain per gallon.

As regarded the composition of the carbonate, there certainly was a large quantity of oxide of zinc present in the deposit, but ordinary carbonate of zinc, would, without doubt, have been decomposed by the high temperature and pressure to which the deposit had in all probability been subjected.

THE COMPOSITION OF HUMAN FAT.

By C. A. MITCHELL, B.A.

(*Read at the Meeting, May 6, 1896.*)

ALTHOUGH it is not probable that members of this Society will be frequently called upon to make an analysis of human fat, yet one can easily conceive the possibility of such a case occurring, and I have therefore thought that, apart from their purely scientific interest, my figures might have a practical value.

Human fat appears to have been but little studied since the days of Chevreul and Heintz—at least, no description of its characteristics is to be found even in books

* This has since been done in a fresh sample, and the water found to be slightly acid. The analysis of this sample showed the following constituents:

						Grains per gallon.
Total solids	6.160
Chlorine	...					0.960
Nitric acid						traces
Oxygen absorbed						0.022
Ammonia	...					none.

dealing specially with physiological chemistry. Chevreul* found that it consisted of an olein and stearin, the latter being distinguished from mutton stearin by yielding "margaric," and not stearic acid. Heintz† came to the conclusion that the composition of the fat was of a more complex nature, and that, in addition to olein and palmitin, there were small quantities of several other acids, one of them being a liquid acid.

The specimen of fat which I have examined, and for which I am indebted to the kindness of Dr. Arnold Chaplin, was separated from the tissue at as low a temperature as possible. It was of a pale yellow colour, and had a consistency somewhat less than that of butter. On crystallization from ether there was only a slight deposit, which, when washed with ether and dried, melted at 51.5° C. Under the microscope the crystals had some resemblance to those frequently obtained from flare lards, being arranged in fan-shaped bunches, while the individual crystals had the characteristic chisel-shaped ends.

The determination of the chemical and physical constants of the fat gave the following figures :

FAT.				
Specific Gravity at 25° C. (Water at 25° = 1)	0.9033
Melting-point	17.5°
Solidification-point	15°
Saponification No.	195
= Equivalent	287
Reichert Value (2.5 grammes)	0.3 $\frac{N}{10}$ alkali.
Acidity No. (1 gramme)	6.3 mgs. KOH = 0.32 per cent oleic acid.
Bromine Heat Value	11.3° C.
= Iodine Value, 11.3×5.5	62.15
Iodine Value (Hübl)	61.5

FATTY ACIDS.				
Melting-point	35.5°
Solidification-point	30.5°
Iodine Value	64
Iodine Value of Liquid Acids	92.1

In order to obtain some idea as to the nature of the fatty acids, Twitchell's modification of Jean's method of fractional precipitation was employed (ANALYST, xx., p. 165). 1.9478 grammes of the fatty acids (iodine number 64) were dissolved in 25 c.c. of 95 per cent. alcohol; 1.5 grammes of lead acetate dissolved in 10 c.c. of the same alcohol were added, and the flask well corked, allowed to stand over-night. In the morning some of the liquid was filtered, the acids recovered by shaking with ether and hydrochloric acid, and their iodine number determined. The precipitate was well washed with alcohol, the fatty acids recovered, dried, and weighed, and their iodine number also determined. In each case precautions were taken to prevent oxidation. The results obtained were :

	Grammes.	Iodine No.	Per cent.
Solid Acids	0.9779	37	50.2
Liquid Acid (by difference)	0.9699	92	49.8

* *Recherches sur les Corps Gras*, 1823.

† *Pogg. Annal.*, 1851, p. 238.

The acids from the filtrate were liquid at 15° C., while those from the precipitate melted at 35.5° C., and had a molecular equivalent of 278.

From these figures the amount of liquid and solid acids can be calculated.

Liquid Acids in filtrate	Per cent.
" " precipitate reckoned as Oleic Acid	49.8
	37×50.2	18.57				20.6
	$\frac{100}{100}$	$= \frac{0.9}{0.9}$				
Total Liquid Acids	70.4
With Iodine No.	$\frac{64 \times 100}{70.4}$	$=$	90.9

From this it appears that the liquid acids probably consist of oleic acid, with a small amount of a more unsaturated acid, possibly linolic.

With regard to the solid acids, the high molecular equivalent (278) of the lead precipitate, of which only 20 per cent. is accounted for by the liquid acids, leads to the conclusion that stearic acid, or some other acid of high molecular weight, is present.

Further evidence of the composition of the solid acids was obtained by crystallizing the fatty acids from 70 per cent. alcohol. The crystals, after being well washed with the same alcohol and dried, melted at 50.5° C. This melting-point being lower than that of any mixture of stearic and palmitic acid in Heintz's table, points to the presence of myristic acid, since the precipitate was quite free from liquid acids. The filtrate, on standing, yielded a second deposit, which also melted at 50.5° C.

Human fat, therefore, would seem to consist of about 70 per cent. of liquid acids, principally oleic acid, 30 per cent. of solid acids, probably palmitic with small amounts of stearic and myristic acids, and traces of lower volatile acids.

DISCUSSION.

The PRESIDENT said that Mr. Mitchell's figures were very valuable, and he hoped to see them incorporated in future text-books on physiology, in supersession of the very imperfect data at present available on the subject of human fat. The fact that the results now brought forward were perfectly novel, to a certain extent prevented discussion of them; but they were certainly worthy of consideration, and credit was due to Mr. Mitchell for bringing them forward.

Mr. HEHNER said he would like to ask Mr. Mitchell for some further explanation of the grounds upon which he assumed that the fluid fatty acids consisted chiefly of oleic acid, with a small amount of linolic acid. He (Mr. Mitchell) had, by means of their lead salts, divided the acids into liquid and solid. He (Mr. Hehner) had shown to the Society that the separation of the lead salts by means of ether was at most but a partial one, and this was borne out by Mr. Mitchell's figures. For instance, the "solid" fatty acids having an iodine absorption of 37 per cent., should not, theoretically, have had any iodine absorption at all, if the process of separation had worked perfectly; and from the fact that the original fat had an iodine absorption of 61, it would seem that separation had really been effected very imperfectly. Certainly the "liquid" fatty acids had an iodine absorption of 92, practically that of oleic acid, but was it not possible that these liquid fatty acids contained a considerable quantity of

the solid acids? At all events, this was the conclusion which he (Mr. Hehner) had arrived at from his own investigations in connection with this process of separation. With lards, to which human fat appeared to have a great resemblance, this process seemed also to give very variable results. The figures which Dr. Muter had published in this connection all showed iodine absorptions of about 90 or 91 for the fluid acids, but further investigation had shown that, from American lard, at all events, much higher figures were obtained, indicating that there must have been a considerable quantity of another fatty acid besides oleic acid, and, considering that solid saturated fatty acids were undoubtedly included in the fluid ones, the amount of the unsaturated fatty acid would be larger than appeared from the iodine absorption. He therefore did not think it followed that because a liquid fatty acid obtained in this way had an iodine absorption of about that of oleic acid, that oleic acid was necessarily present.

Mr. BEVAN inquired what part of the body the fat had been taken from. In the case of the pig, the lard varied very considerably, according to the part of the body from which it was obtained. Also whether Mr. Mitchell had determined the Valenta number, and the refractive index in the oleorefractometer.

Mr. MITCHELL, referring to the question of the iodine absorption of the liquid fatty acids, said that no system of identification which depended simply upon the iodine absorption test could be regarded as conclusive, but that since, so far as he was aware, no acid absorbing less than 90 per cent. of iodine had yet been found in fats, he considered it probable that when an iodine number of about 90 was obtained in the liquid acids prepared by Twitchell's method it represented oleic acid. Twitchell had shown that in his process of separation there was a fractional precipitation, the lead salt of oleic acid being precipitated first, then that of linolic, and finally that of linolenic acid. He therefore calculated any iodine absorption in his precipitate as oleic acid. Since by this fractionation process he (Mr. Mitchell) had found that the acids from the filtrate only absorbed 92 per cent. of iodine, whereas Twitchell frequently had an iodine absorption of over 100, it was highly probable that acids more unsaturated than oleic acid were only present in small quantities. At the same time, the data were insufficient for anything more than a probable conjecture. He believed that the fat he had examined was from the kidneys. He had not determined either the Valenta number or the refractive index.

ON THE COMPOSITION AND ANALYSIS OF COMMERCIAL CREAM OF TARTAR.

By ALFRED H. ALLEN.

(Read at the Meeting, June 3, 1896.)

WHEN prepared by boiling crude tartar or "argol" with water, filtering, and crystallizing the salt from the clear liquid, cream of tartar always contains more or less calcium tartrate, which, though nearly insoluble in cold water, dissolves with moderate facility in a hot solution of acid tartrate of potassium. In a paper read by

me before this Society in April, 1880, I described some experiments made to ascertain the extent to which calcium tartrate could occur unavoidably or legitimately in cream of tartar. To avoid reference, I may recapitulate the nature and results of these experiments, as described in *THE ANALYST*, vol. v., p. 114.

Pure calcium tartrate was prepared by precipitating calcium chloride by neutral sodium tartrate prepared from pure tartaric acid and sodium carbonate. The analysis of the salt showed that it was strictly neutral in composition.

Pure potassium hydrogen tartrate was prepared by dividing a solution of pure tartaric acid into two equal portions, neutralizing one with potassium carbonate, and adding the other.

Weighed quantities of the last product were dissolved in known measures of boiling water, an excess of moist tartrate of calcium added, the liquid boiled for a short time, filtered boiling hot, and the crystals of cream of tartar deposited on the cooling of the filtrate were analysed. In this manner, products were obtained which contained as much calcium tartrate as would dissolve in the measure of boiling solution of tartrate of potassium employed. The only variable condition was the proportion of water used.

It was not found practicable to dissolve the acid tartrate of potassium in fifteen times its weight of boiling water. Hence, one in twenty-five was the strongest solution employed. The following are the percentages of insoluble ash and anhydrous neutral calcium tartrate contained in the different products :

Proportion of Water used.				Insoluble Ash.		$\times 1.88 = \text{CaC}_4\text{H}_4\text{O}_6$.
1.	...	25 parts	...	3.10 per cent.	...	5.82 per cent.
1 A.	...	25 "	...	3.28 "	...	6.16
2.	...	50	...	3.40	...	6.39
2 A.	...	50	...	3.30	...	6.20
3.	...	75	...	4.40	...	8.27
3 A.	...	75	...	4.80	...	9.02

From these results it appears that the proportion of calcium tartrate contained in cream of tartar is greater the larger the proportion of water employed for solution ; but that, with the quantity of water likely to be used in practice, the product will not contain more than nine or ten per cent. of calcium tartrate. This is fully confirmed by the experience of myself and others as to the composition of commercial cream of tartar. One or two per cent. in excess of ten may be allowed by the Public Analyst as a margin, but it may safely be concluded that any sensibly higher proportion of calcium tartrate is not a normal constituent of the sample. Cases have occurred in which crude tartar or argol has been substituted for cream of tartar, which is a product refined by crystallization from water. In crude tartar the percentage of calcium tartrate found may be very high, especially if it be derived from plastered wine ; but in such cases more or less sulphates will also be present. In a sample of cream of tartar submitted to me some years since I found as much as 20 per cent. of calcium sulphate. This was not crystallized, as it would have been if derived from the plastering process, but had apparently been intentionally added as an adulterant, probably in the form of plaster of Paris.

According to the British Pharmacopœia of 1867, "188 grains (of cream of tartar)

heated to redness till gas ceases to be evolved, leave an alkaline residue, which requires for exact neutralization 1,000 grain measures of the volumetric solution of oxalic acid." Nothing is said here about operating on the filtered extract of the ash, so that the neutralizing power of the calcium carbonate derived from the calcium tartrate would be included. As a matter of fact, 188.1 grains of pure potassium hydrogen tartrate are required to neutralize 1,000 grain-measures of normal oxalic acid, so that the test leaves no room for any impurity whatever, except calcium tartrate, and the larger the proportion of this substance there is present the better the sample will appear. By a coincidence, the molecular weights of KHT and CaT are identical, but the residue left on igniting the latter will have twice the neutralizing power of that yielded by the former. Hence the residue from 188 grains of calcium tartrate would neutralize 2,000 grain-measures of normal acid.

In the British Pharmacopœia of 1885, the foregoing test for the purity of cream of tartar was modified, the directions being as follows:—"Dried on a water-bath, 204 grains, heated to redness till gas ceases to be evolved, leaves an alkaline residue, which, when treated with distilled water, filtered, and well washed, yields a clear solution requiring for exact neutralization at least 1,000 grain-measures of the volumetric solution of oxalic acid." This amount of acid corresponds to 92.15 per cent. of potassium hydrogen tartrate.

Most analysts will avoid using the clumsy amount of the sample prescribed in the British Pharmacopœia, and will operate on 5 grammes, which, when ignited, should give a soluble extract which neutralizes not less than 24.4 c.c. of normal acid. It will be observed that the British Pharmacopœia test allows less than 8 per cent. for calcium tartrate and all other impurities.

In a recent case at Belfast in which a vendor of cream of tartar was prosecuted on the certificate of Sir Charles Cameron for selling an article adulterated with 17.93 per cent. of tartrate of lime, Mr. S. Templeton, F.I.C., of the Belfast School of Applied Chemistry, was called for the defence. In the face of the very precise standard of purity adopted by the Pharmacopœia, Mr. Templeton stated that the British Pharmacopœia allowed a variable quantity of calcium tartrate up to about 15 per cent., and that he would consider cream of tartar containing that proportion as a fairly good commercial article! When taxed with this statement in the Belfast press Mr. Templeton argued that the Pharmacopœia test only refers to 20.9 per cent. of the radical potassium, and that it is not justifiable to assume that all this is present as acid potassium tartrate. Mr. Templeton further suggests that some may be present as neutral potassium tartrate, but he does not explain how he would account for the presence of any notable quantity of such an extremely soluble salt in an article prepared after the manner of cream of tartar; and, unfortunately for Mr. Templeton's argument, the British Pharmacopœia test includes any neutral potassium tartrate, and credits it as bitartrate! Not only does it do this, but 116 parts of K_2T produce on ignition as much K_2CO_3 as is yielded by 188 of KHT ; so that if a sample consisted wholly of neutral potassium tartrate it would show by the British Pharmacopœia test 162 per cent. of acid tartrate!

The character of commercial cream of tartar has materially improved of late years, and an article which might be tolerated in 1884, when vol. i. of my "Com-

mercial Organic Analysis" appeared, would now be regarded as outside the pale. This is evident from the following facts.

In the United States Pharmacopœia of 1885, 6 per cent. of impurity was permitted in cream of tartar, but in the edition of 1890 the standard of purity was raised to 99 per cent., as ascertained by titrating the solution of the original sample with standard alkali, using phenolphthalein as the indicator.

The German and Japanese Pharmacopœias also fix the standard of purity for cream of tartar at 99 per cent.

In 1888 (*Pharm. Jour.* [3], xviii., 1045) Messrs. Southall Brothers and Barclay published the results of the assay by Mr. R. A. Cripps, by the British Pharmacopœia process, of sixty-eight samples of commercial cream of tartar. Of these, seventeen were found to answer the requirements of the British Pharmacopœia, but almost without exception a sample taken after grinding the bulk gave a lower result than a sample taken from the top of a cask, as is done when sampled by a broker. Of the casks the samples from which answered the British Pharmacopœia test twelve were purchased, but the contents of these when powdered reached the official standard in three cases only.

In a paper read before the British Pharmaceutical Conference at Leeds in 1890, Mr. H. Broadbent gave the following figures obtained by the analysis of cream of tartar:—

	Italian.	French.	German.
Number of samples contributing to average ...	40	6	6
Average percentage composition:—			
Acid potassium tartrate	94.11	93.00	93.05
Neutral potassium tartrate	1.52	1.73	1.45
Calcium tartrate	4.02	4.78	5.00
Insoluble matter	0.23	0.28	0.25
Moisture	0.27	0.30	0.32

The majority of those who took part in the discussion of Mr. Broadbent's paper were of opinion that the British Pharmacopœia requirement of 92.15 per cent. on the moisture-free article was not too high.

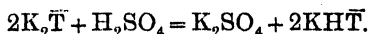
In a paper read by Mr. T. A. Ellwood in 1891 (*Pharm. Jour.* [3], xxii., 392), he stated that he had never found more than 6.5 per cent. of calcium tartrate in cream of tartar, and was of opinion that the Pharmacopœia might advantageously limit the amount of this impurity to 4 per cent. In a protest against this suggestion (*ibid.*, page 448) Mr. R. A. Cripps quoted the results obtained by him in 1888 and already mentioned.

Quite recently Mr. M. Conroy has expressed the opinion that the British Pharmacopœia standard should be raised to at least 96 per cent., and Messrs. Kirkpatrick, Barr and Guthrie, a leading firm of importers, who were the first to raise the trade-test to 95 per cent. independent of the British Pharmacopœia, express the opinion that the standard should be raised to 99 to 100 per cent., as in other countries (*Pharm. Jour.* [4], ii., 346, 420).

It is evident, therefore, that it is very generally considered that the existing British Pharmacopœia standard of purity is quite low enough, and that there is no

excuse for the sale, under the name of cream of tartar, of such an article as formed the subject of the recent proceedings at Belfast.

Of late years a good deal of cream of tartar has appeared on the market which has been prepared by a method essentially different from that hitherto considered. It appears to have been produced under one of the modifications of the patent of A. Martignier (No. 20,078, of 1889). According to this process, neutralized wine-lees are treated with an excess of a saturated solution of potassium sulphate, whereby calcium sulphate and neutral potassium tartrate are formed. The gypsum is separated, and the solution of potassium tartrate decolorized with animal charcoal and treated with sulphuric acid in quantity sufficient for the reaction :—



It is evident that an article manufactured in this manner will be free from calcium tartrate, but will be liable to contain sulphates of potassium and calcium. In fact, much of the cream of tartar of commerce now contains several units per cent. of calcium sulphate, just as an article made in the old way contained more or less calcium tartrate as a normal impurity. Both impurities are perfectly neutral, useless and harmless, so far as the consumer is concerned, but with a little trouble they both could, and would, be removed, if the present British Pharmacopœia test were raised to the standard adopted in some other countries.

I made a very complete examination some years since of a sample of cream of tartar prepared by the sulphuric acid process, and which yielded no trace of free sulphuric and tartaric acid to alcohol. I adopted the following line of reasoning as to the normal constituents of cream of tartar.

The acidity of the sample may be due to :—

- a. Potassium acid tartrate, KHT .
- b. Potassium acid sulphate, KHSO_4 .

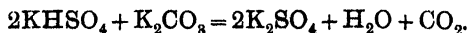
The neutral salts which may possibly be present are :

- c. Calcium tartrate, CaT .
- d. Potassium sulphate, K_2SO_4 .
- e. Calcium sulphate, CaSO_4 .
- f. Neutral potassium tartrate, K_2T .

Of course, no neutral potassium tartrate can possibly be present in an article crystallized from water containing sulphuric acid, and I very much doubt its occurrence in cream of tartar manufactured by the old process.

On ignition, the following changes would occur :—

- (a) KHT would yield K_2CO_3 in amount just corresponding with the original tartrate ; so that one gramme of ignited KHT would exactly neutralize one gramme of unignited KHT .
- (b) Any acid sulphate of potassium would react with the K_2CO_3 thus :—



In practice this would occur during the ignition. Any K_2SO_4 produced is neutral, and hence any KHSO_4 originally present will reduce the alkalinity of the ash. It does this not only by not yielding alkalinity itself, as KHT would do, but also by neutralizing an equivalent amount

of K_2CO_3 from the tartrate. Hence, if a sample contained KHT and $KHSO_4$ in equivalent proportions, on ignition it would give only K_2SO_4 , and the ash would not be alkaline.

Only trifling quantities of acid sulphate of potassium are likely to occur as a normal impurity in cream of tartar.

- (c) CaT on ignition yields $CaCO_3$, and hence the ignited sample will neutralize a proportionate quantity of acid. But $CaCO_3$ being nearly insoluble in aqueous liquids, the portion of the ash soluble in water will be deficient in acid-neutralizing power as the CaT increases. But if the insoluble ash be included in the titration, the neutralizing power will exceed the acidity of the original sample.
- (d) K_2SO_4 is not affected by ignition, and hence may be left out of consideration.
- (e) $CaSO_4$ is not affected by ignition, but will react with the K_2CO_3 (derived from the KHT) thus:— $CaSO_4 + K_2CO_3 = CaCO_3 + K_2SO_4$.

Hence calcium sulphate will reduce the alkalinity of the aqueous solution of the ash (and so cause a low British Pharmacopœia test), but the acid-neutralizing power of the *total* ash will not be affected.

- (f) K_2T will burn to K_2CO_3 , and the ash will have a higher neutralizing power than that yielded by an equal weight of KHT .

From these considerations it follows that if a sample of cream of tartar be free from uncombined sulphuric and tartaric acids, and contains only calcium and neutral potassium sulphates and potassium acid tartrate, it will before ignition neutralize a volume of standard alkali exactly equivalent to the acid required to neutralize the sample after ignition, including the alkalinity of the insoluble ash.

If $KHSO_4$ be present, the acidity of the original sample will be *in excess* of the total alkalinity of the ash. If CaT be present, the acidity of the sample will be *less* than the alkalinity (total) of the ash. As $KHSO_4$ and CaT are incompatible in solution, both impurities cannot be present in the same sample of *genuine* cream of tartar. In an article prepared by crystallization from water, and subsequently adulterated with $KHSO_4$ (which has been actually used as a substitute for cream of tartar, under the name of "tartaline"), the two salts might co-occur.*

A sample containing neutral potassium tartrate would yield on ignition an ash having a greater acid-neutralizing power than the acidity of the original cream of tartar.

It is evident that when applied to samples of cream of tartar containing calcium sulphate, the existing British Pharmacopœia test will indicate a proportion of acid potassium tartrate materially below the truth. Every unit of (anhydrous) calcium tartrate present will reduce the indication of acid potassium tartrate by 2.77 per cent., so that a sample containing only 4 per cent. of $CaSO_4$ will show by the British Pharmacopœia method only 88.92 per cent. of acid tartrate. Such a result is manifestly intolerable, and it is to be hoped that the next edition of the British Pharmacopœia will abolish this anomaly, and place the tests for cream of tartar on a level with those adopted in other countries. I may say that I have communicated with Dr.

* As acid potassium sulphate is deliquescent, its presence in cream of tartar as an intentional adulterant is improbable. On the other hand, I have found notable traces in an article prepared by the acid process.

Attfield on the subject, but he declined to furnish any indication of what might be expected, though in his own practice he favours direct titration.

It is, of course, important that the Pharmacopœia methods should be as simple as possible, but the existing test for cream of tartar scarcely complies with this requirement. A direct titration of the original sample with standard caustic alkali and phenolphthalein would be preferable in every respect, if adulteration by acid potassium sulphate were prevented by fixing a limit to the amount of sulphates admissible. Where further information is required, it is readily obtainable by noting the acid-neutralizing power of the soluble and insoluble ash, and comparing them with the acidity of the original sample as already described. For this purpose I would propose the following process:—

1. Dissolve 1·881 grammes of the moisture-free sample in hot water and titrate with decinormal caustic alkali and phenolphthalein. In the absence of acid potassium sulphate (and of free tartaric acid), each c.c. of alkali required represents 1 per cent. of acid potassium tartrate in the sample.

2. Ignite 1·881 grammes of the moisture-free sample at a dull red heat for ten minutes, without attempting to burn off all the carbon. Boil the product with water, filter, and wash the insoluble carbonaceous residue.

(a) Titrate the filtrate with decinormal hydrochloric acid and methyl-orange. In a pure sample the measure of acid required will be exactly equal to that of the alkali consumed in process 1. Each c.c. of *deficiency* represents 0·50 per cent. of calcium tartrate, $\text{CaC}_4\text{H}_4\text{O}_6$; 0·36 per cent. of calcium sulphate, CaSO_4 ; or 0·60 per cent. of neutral potassium tartrate. Any *excess* points to the presence of acid potassium sulphate KHSO_4 , each c.c. of difference representing 0·71 per cent. of that salt. If the titrated liquid be treated with barium chloride, the weight of the precipitate of barium sulphate obtained will give the means of directly determining the proportion of calcium or potassium sulphate, and of deducing the calcium tartrate.

(b) Ignite the carbonaceous residue, dissolve in 20 c.c. of decinormal acid, filter if necessary from any insoluble residue of sand, barium sulphate, etc., wash, and titrate the filtrate with decinormal alkali and methyl-orange. The measure required represents the calcium of the sample. Each c.c. corresponds to 0·50 per cent. of calcium tartrate, or 0·36 per cent. of calcium sulphate (anhydrous).

The quantity of cream of tartar directed to be employed in the foregoing process is somewhat smaller than is desirable. Hence, in cases of importance, it would be preferable to operate on a double quantity, that is 3·762 grammes. Of course, the percentages of constituents corresponding to each c.c. of decinormal acid and alkali would, in this case, be one-half of the amounts stated above.

In the above process, I have followed the direction of the British Pharmacopœia to operate on the moisture-free sample, but in practice it would be easier to analyse the original sample, and determine the moisture on a separate portion, making any allowance that might be necessary.

In exceptional cases, it may be necessary to examine cream of tartar by determining the total tartaric acid, but the analysis of such samples is outside the scope of this paper.

DISCUSSION.

The PRESIDENT said that some years ago he had analysed a large number of samples of commercial cream of tartar, and had found that as a rule they contained from 89 to 92 per cent. of the pure salt, as tested by their acidity.

He was a little at a loss to understand how a sample of cream of tartar could normally contain potassium bisulphate. It seemed to him that the presence of both bisulphate and bitartrate would give rise to the presence of free tartaric acid. It appeared to be a matter of discussion whether a sample of genuine cream of tartar really could contain any sulphuric acid in the form of a bisulphate.

Sulphate of lime used to be commonly found in samples of cream of tartar, and also very commonly barium compounds in small quantities. These barium compounds were in an absolutely insoluble form, but in such small quantities that it was difficult to imagine them to have been added intentionally.

He was very glad to hear that pure, or almost pure, bitartrate of potassium was an article of commerce. It was not very many years ago that he had experienced very great difficulty in getting a single pound prepared containing, say, 97 per cent. of real bitartrate of potassium.

Sir CHAS. A. CAMERON said that the specimen referred to by Mr. Allen was the worst specimen of cream of tartar on sale in Ireland that he had ever examined. A great many samples had been examined in his laboratory, and the general average was fairly good. He might say, he was rather surprised that a member of his own profession should regard as commercially pure an article of which nearly one-fifth consisted of a foreign ingredient. Since the publication in the Belfast papers of the correspondence referred to, several manufacturers had written to him, sending samples of cream of tartar, and stating that they had for a considerable time been putting on the market an article containing 96 per cent. of pure cream of tartar.

Dr. DYER said he had examined a great many samples of cream of tartar, and it was quite a common thing for them to contain 96 per cent. and more of the pure article. He remembered two samples which contained 98 per cent. Samples of such high quality always contained sulphate of potash, and were generally quite free from lime.

He had often heard complaints of darkening taking place in cream of tartar. This generally occurred in samples made by the sulphate process, and was evidently due to traces of iron forming traces of sulphide when, through organic decomposition, the sulphates became "reduced."

He would like to ask Mr. Allen whether he had had any experience of the going bad of cream of tartar by clogging and forming lumps. On several occasions samples had been submitted to him of cream of tartar which had arrived after a voyage to Australia in this condition, and he had invariably found an enormous quantity of fungus running completely through the material and matting it together. The percentage of moisture was usually small, and it would be interesting to know what were the actual circumstances in packing or storing that determined this free fungoid growth.

Mr. HEHNER suggested that the presence of barium compounds might possibly be accounted for in the following way: All Southern wines used to be very heavily

plastered, which converted the acid potassium tartrate into calcium tartrate and potassium sulphate. This was done partly because the colour of the wine was thereby heightened, and partly because the precipitate of calcium tartrate carried down any suspended impurities along with it, leaving the wine clear. Excessive plastering, however, and the consequent presence of a large amount of potassium sulphate in solution, were said to have very baneful effects, and several processes had been devised with the object of converting the potassium sulphate into potassium tartrate again. This was generally effected by means of barium tartrate, or barium hydrate and tartaric acid, the result being the re-formation of the acid potassium tartrate, together with a precipitate of barium sulphate.

An edict had now been passed in France regulating the practice of plastering, and limiting the proportion of sulphates to 2 grammes per litre, calculated as K_2SO_4 .

Mr. ALLEN said that if barium sulphate was in any way a natural constituent of cream of tartar, it would certainly be present in an amorphous form. It had, however, been found in the form of crystallized heavy spar, which clearly showed that it had been fraudulently added. Besides, for some years past it had entirely disappeared.

With regard to the acid sulphate of potassium, he agreed with the President that it was a very unlikely constituent of cream of tartar, in notable quantity, unless purposely added. It had, however, been used as a substitute for cream of tartar under the name of tartaline, so that its occurrence as an adulterant of that material seemed not altogether impossible.

It was known that fungoid growths were liable to develop in cream of tartar, but he had always imagined this to be due to excess of moisture. The low percentage of moisture in the cases referred to by Dr. Dyer negatived such a conclusion in this instance, and he was quite unable to offer any other explanation of the phenomenon.

He had in his possession a letter, written as long ago as in 1893, stating that there were five factories in America turning out weekly, in the aggregate, 200 tons of bitartrate containing 99 to 100 per cent. of the pure salt, which was good evidence that it could be obtained on a large scale. But there was no encouragement for manufacturers to place a superior article on the market, while the British Pharmacopœia requirement was so much below that of other countries and the official test in certain cases gave a result materially below the truth.

DETERMINATION OF LACTOSE IN MILKS BY DOUBLE DILUTION AND POLARIZATION.

H. W. WILEY AND E. E. EWELL.

In vol. vi., p. 289, of the *American Chemical Journal*, one of us (Wiley) published an article on the determination of lactose in milks by optical methods. The principal novelty in this process was the substitution of mercuric nitrate as the reagent for precipitating proteids in place of the other reagents which had usually been employed for that purpose. By the use of mercuric nitrate in an acid solution, it was shown

in that paper that it was possible to practically throw out all of the proteids dissolved in the milk. Inasmuch as these soluble proteids are optically active, and deflect the plane of polarization in a direction opposite to that produced by lactose, the presence of any notable quantity of them in the solution to be polarized tends to diminish the apparent percentage of lactose present. The reagent proposed, viz., acid mercuric nitrate, when used in the cold and in the quantities specified, produces no inversion effect whatever upon the lactose.

In the paper referred to, an arbitrary correction was made for the volume of the precipitate produced, and this was fixed at 2.5 c.c., when approximately 60 c.c. of milk were used in a 100 c.c. flask.

This method of estimating lactose, on account of the ease with which it can be operated and its accuracy, has been generally adopted by chemists. Attention has been called, however, to the fact that the arbitrary correction allowed for the volume of the precipitate is too small. (See ANALYST, vol. xii., p. 64; vol. xx., p. 126.)

Theoretically, it is evident also that the arbitrary correction admitted is too small except in cases of well-skimmed milk. In order to eliminate this arbitrary factor from the method, we undertook a series of experiments to determine the actual percentage of sugar and the proper correction to be allowed for the volume of the precipitate by the method of double dilution and polarization originally proposed by Scheibler for sugar solutions, and suggested by Bigelow and McElroy for use in the polarization of milk sugar. (*Journal of the American Chemical Society*, xv., 694.) The results of our determinations are extremely satisfactory, and show that the volume which is occupied by the precipitate in a milk varies from 2.5 c.c. in the circumstances mentioned above, to 6 c.c., according to the richness of the milk in fat. It appears, however, that this correction is less in quantity than the combined volume of the fat and albuminoids, which may be safely assumed to be 1 c.c. for 1 gramme.

All the flasks which were employed in the determinations were carefully calibrated, and the instrument used was the new triple-field shadow polariscope, made by Schmidt and Haensch, which enables readings to be accurately made to within 0.05 of 1 per cent. All readings were made in duplicate by each of us and entered before comparisons were made, and in the polarizations given in the table the means of these four readings, which never differed by more than one-tenth of 1 per cent., are given. The polarizations were made on the contents of a 100 and a 200 c.c. flask, after clarification of the milk by means of acid mercuric nitrate. In each case, double the quantity of the normal weight of milk for the instrument used was taken. The readings were calculated by the formula given by Scheibler, which requires that the reading obtained from the solution in the large flask be multiplied by 2 and subtracted from the reading obtained in the small flask. In all cases, in order to secure greater accuracy, our readings were made in a tube 400 millimetres in length. Therefore the data obtained in reading the solution in the small flask were divided by 4 in order to obtain the apparent percentage of sucrose.

The application of the formula given by Scheibler does not give absolutely accurate results. The true polarization in any given case is calculated according to the following scheme:

Let x equal the volume of the precipitate and y the correct reading. Let a equal

the reading obtained from the solution in the small flask and b equal the reading of the solution from the large flask. We then have :

$$\begin{aligned} 200 - x : 100 - x &:: a : b \\ 200b - bx &= 100a - ax \\ ax - bx &= 100a - 200b \\ x &= 100 \frac{(a - 2b)}{a - b} \quad (1) \end{aligned}$$

$$\begin{aligned} 100 - x : 100 &:: y : a \\ 100 - 100 \frac{(a - 2b)}{a - b} &: 100 :: y : a \end{aligned}$$

$$\begin{aligned} 100 \left(a - \frac{a(a - 2b)}{a - b} \right) &= 100y \\ y &= a - \frac{a(a - 2b)}{a - b} \\ y &= \frac{a^2 - ab - a^2 + 2ab}{a - b} = \frac{ab}{a - b} \quad (2) \end{aligned}$$

The rule derived from formula No. 2 is as follows :

The true polarization, as determined by double dilution, is found by dividing the product of the two readings made from the solutions in the large and small flasks by their difference.

In order to test the accuracy of the method, known volumes of insoluble material, as, for instance, quartz sand, were added to the flasks in order that the volume of the precipitate might be increased by a certain definite amount. The determinations were also made on the whole milk as purchased, on the same milk deprived of the most of its cream, and on the cream thus secured. In all cases the results obtained were satisfactory.

Blyth has recently described a method of precipitating the casein with acetic acid, and of washing the precipitate free from sugar on a filter, and polarizing the filtrate (*ANALYST*, vol. xx., p. 122). The percentage of milk-sugar in the mixed filtrate and washings is about one and the polariscopic reading should be corrected for that degree of solution. This method evidently is better suited for preparing milk whey for the gravimetric estimation of the sugar by copper, since it takes no account of the albumins left in solution, which serve to a certain extent to counteract the polarizing power of the lactose.

In the presence of sucrose he proposed to estimate its quantity from the property possessed by citric acid of inverting the sucrose and leaving the lactose unchanged.

Raumer and Späth (*Zeit. Angew. Chem.*, 1896, p. 72) suggest that the polarization of milk should be preceded by boiling, since it is probable that the lactose may exhibit birotation. The data which they adduce, however, are far from convincing, since, after the boiling, they clear the mixture with lead subacetate, and it has been shown that this reagent does not remove all the proteids. The deficit in rotation is therefore probably due to the residual soluble left-handed proteids. They further suggest that the presence of a dextrinoid body, as indicated by Ritthausen (*Journ. Prakt. Chem.*, (2), xv. 348) may serve to increase the actual rotation of the

milk-sugar. In the samples which showed the apparent increase, however, they made no attempt to prove the presence of the alleged disturbing dextrin.

There seems to be no just reason, therefore, for insisting on the slow and tedious gravimetric method when a quick and accurate optical method is at hand.

Inasmuch as the time required for carrying out the method of double dilution and polarization is scarcely any longer than that required for a single polarization, it is recommended that it be done in all cases, instead of correcting the results of a single polarization by any arbitrary factor. When the determination is conducted as suggested the analyst has at hand an easy, rapid, and accurate method of estimating milk-sugar in milk, which is as desirable in all respects as any gravimetric method whatever. The data obtained are given in the accompanying table.

POLARIZATION OF MILK BY DOUBLE DILUTION.

No.	Per cent. Fat.	Polarization in 200 c.c. Flask.	Polarization in 100 c.c. Flask.	Apparent per cent. Lactose.	True per cent. Lactose.	True volume in 100 c.c. Flask.	Volume of Precipitate.
1		9.37	19.26	4.82	4.56	94.4	5.6 ¹
2		9.59	20.33	5.08	4.54	88.8	11.6 ²
3		9.36	19.20	4.80	4.57	95.0	5.0 ³
4		9.60	20.25	5.06	4.56	89.7	10.3 ⁴
5	2.9	10.15	20.84	5.21	4.95	94.8	5.2
6	4.8	10.31	21.21	5.30	5.00	94.5	5.5
7	3.1	9.49	19.41	4.85	4.64	95.7	4.3 ⁵
8	4.0	10.01	20.45	5.11	4.90	95.9	4.1
9	1.4	9.44	19.26	4.82	4.63	96.1	3.9 ⁶
10	5.5	11.05	22.68	5.67	5.38	94.8	5.2
11	4.4	9.57	19.47	4.87	4.71	96.5	3.5 ⁷
12	2.0	9.75	19.93	4.98	4.77	95.8	4.2 ⁸
13	17.6	8.72	19.13	4.78	4.01	82.4	17.6 ⁹

¹ Without sand.

² With 5 c.c. quartz sand.

³ Without sand.

⁴ With 5 c.c. quartz sand.

⁵ Same as No. 6, after separation of cream.

⁶ Same as No. 8, after separation of cream.

⁸ Skimmed milk.

⁹ Cream.

⁷ Whole milk.

Summary of Method.—For the scale of the instrument used, 32.91 grammes of pure lactose in 100 c.c. give a reading of 100. This number is derived from the following data: For sucrose, concentration 26 grammes in 100 c.c., $[\alpha]_D^{20} = 66.37$. For lactose, 33 grammes in 100 c.c., $[\alpha]_D^{20} = 52.53$; then $66.37 : 52.53 :: x : 26.048$; whence, $x = 32.91$. The temperature of the working room should be kept at about 20° C., since the rotatory power of lactose rapidly diminishes as the temperature rises. Double this quantity, viz., 65.82 grammes of milk, is placed in a 100 c.c. flask, clarified with mercuric nitrate solution, the volume completed to the mark, the contents of the flask well shaken, poured upon a filter, and the filtrate polarized in a 400 mm. tube. A similar quantity of the milk is placed in a 200 c.c. flask, and subjected to the same treatment. The polarimetric data obtained are used for calculating the true volume of the liquid in the flask, the true percentage of lactose

and the true volume occupied by the precipitate, in accordance with the rule already given. The acid mercuric nitrate solution is prepared as follows :

Dissolve mercury in double its weight of nitric acid, specific gravity 1.42, and add to the solution five volumes of water. This solution is more dilute than the one recommended in the original paper, it having been noticed that a stronger solution colours the precipitated proteid matter slightly yellow (xanthoproteic reaction). Ten c.c. of the reagent are to be employed instead of two, as directed for the stronger solution. In preparing the solution of milk in the 200 c.c. flask, it may be necessary at times to use more than this quantity of the acid mercuric nitrate, in order to secure a filtrate entirely free of turbidity.

An inspection of the data in the table shows a general agreement between the volume of the precipitate found and the percentage of fat in the sample, with the exception of one instance, viz., No. 11. It is evident that in solutions so dilute a slight variation in the volume has a very small influence on the percentage of sugar found. An error of 0.05° in the reading of the dilute solution (200 c.c. flask) makes an error of 0.05 per cent. in the result. The error due to 1 c.c. of the precipitate in the dilute solution is approximately 0.05 per cent. It is therefore evident that with proper care the percentage of sugar can be determined to within one-tenth of 1 per cent. by the polarimetric method, and this is entirely sufficient for all practical purposes.

CHEMICAL LABORATORY OF THE UNITED STATES DEPARTMENT OF AGRICULTURE.

OFFICIAL METHODS FOR THE ANALYSIS OF FERTILIZERS, ISSUED
BY THE GERMAN MANURE MANUFACTURERS' ASSOCIATION,
HARZBURG, MAY 28, 1895.

CONTRIBUTED BY H. H. B. SHEPHERD, F.I.C., ANGLO-CONTINENTAL GUANO WORKS,
LONDON.

(Continued from p. 156.)

D. *Water Determination.*—In muriate of potash and similar salts the water is determined by heating 10 grammes of the sample in a covered crucible to a dull-red heat for about ten minutes. In the case of salts rich in magnesia, the error due to the decomposition of the magnesium chloride by heat may be avoided by covering the weighed portion in the crucible with a layer of thoroughly burnt lime ; or, the chlorine may be determined by titration before and after heating, and the necessary correction made, taking, of course, into account its replacement by oxygen.

Appendix to Section IV.—(1) *Preparation of Platinic Chloride from the Alcoholic Washings and Precipitates from Potash Determinations.*—The alcoholic washings, diluted with about one-third water, are boiled in a large porcelain basin on the water-bath with sodium carbonate, the potassium platinum chloride precipitates being added a little at a time. The evaporation is continued until the liquid standing over the reduced platinum becomes quite clear and almost colourless. The liquid is then poured off and the reduced platinum purified by boiling with hydrochloric acid and water, after which it is dried and then ignited, to get rid of organic matter. The

platinum-black thus obtained is, before dissolving, rubbed to a fine powder and boiled once with pure concentrated nitric acid, the nitric acid being poured away. Its solution is effected by heating in a large porcelain basin on the water-bath with four times its weight of hydrochloric acid, and adding nitric acid by degrees, in the proportion of 1 part nitric acid to 4 parts hydrochloric acid. When it has completely dissolved, the solution is evaporated until a drop taken out on the end of a glass rod crystallizes. The resulting platinic chloride, which forms into a cake on cooling, is dissolved in water, the solution filtered and adjusted by addition of water so that 10 c.c. of the solution shall contain 1 gramme of metallic platinum (specific gravity 1.18). Special care must be taken that the solution contains neither platinous chloride nor nitrogen oxides. Any platinous chloride can be converted into platinic chloride by treatment with fuming hydrochloric acid and a little nitric acid, and nitrogen compounds can be expelled by alternate evaporations with hydrochloric acid and water. If scrap platinum is used for making the solution, the platinic chloride will be contaminated with a little iridium. This can, however, be separated by precipitating the platinum with ammonium chloride, and then reducing it to the metallic state. The purity of the solution can be most conveniently tested by means of an 80 per cent. muriate of potash sample of known composition, and prepared from pure materials.

(2) *Preparation of Barium Chloride Solution.*—One hundred and twenty-two grammes of crystallized barium chloride are dissolved in water with 50 c.c. of concentrated hydrochloric acid, and the solution made up to 1 litre.

(3) *Alcohol.*—For washing the precipitate, alcohol of at least 96 per cent. must be used.

(4) *Filters.*—Munktell Swedish filters, No. 1 F, are the most suitable for filtering off the potassium platinic chloride.

V. *Determination of Oxide of Iron and Alumina in Phosphates.*—The only method yielding serviceable results is Eugen Glaser's, modified by R. Jones.* Ten grammes of substance are dissolved in 25 c.c. HNO_3 † of 1.2 specific gravity, and the solution made up to 500 c.c. Fifty c.c. of this solution (equal to 1 gramme substance) are evaporated to 25 c.c., and mixed while still warm with 10 c.c. sulphuric acid (1 part concentrated H_2SO_4 to 5 parts H_2O). After stirring, 150 c.c. of alcohol are added, the mixture again stirred and allowed to stand for at least three hours. The calcium sulphate is then filtered off and washed with absolute alcohol, the filtrate being collected in an Erlenmeyer flask of 400 to 500 c.c. capacity. The washing must be continued until 10 drops diluted with an equal quantity of water give no red tint on addition of a drop of solution of methyl-orange. A small Bunsen pump may be usefully employed in the washing. The alcohol is now distilled off, the liquid washed out into a beaker, made feebly alkaline with ammonia, and then heated until all excess of ammonia is expelled; this is an important point, as otherwise the precipitate may contain magnesia. The precipitate is then collected upon a filter, using the ordinary rubber and cold water to remove any that may adhere to

* *Zeitschrift für angewandte Chemie*, 1889, p. 636.

† The use of nitric acid as a solvent in the Glaser process results in the inclusion of iron pyrites as oxide of iron. This is contrary to the English usage. See paper by the Translator, *ANALYST*, vol. xviii., p. 261.—EDITOR.

the sides of the beaker, and washed four times with boiling water, by pouring out of the mouthpiece of the wash-bottle, so as not to disturb the precipitate. In this way clear filtrates can always be obtained. If still greater accuracy is desired, Fresenius's proposal may be adopted, which consists in adding some ammonium nitrate (of course not acid) to the wash-water. The precipitate is finally burnt off and weighed. It is regarded as ferric phosphate and aluminic phosphate, and half its weight taken as oxide of iron and alumina. Should it be desired to weigh the calcium sulphate, it may be transferred while still moist to a platinum crucible, the filter-paper laid over it, the spirit burnt off, and the precipitate and paper ignited at a moderate temperature until the weight remains constant. The calcined calcium sulphate is not too hygroscopic to prevent the use of an open crucible.

It is not needful to resort to Jones's modification, except in cases of dispute. The simple Glaser method, without the evaporation of the phosphate solution or the washing of the calcium sulphate—merely filling up to the mark—gives sufficiently accurate results, and is much more rapidly carried out.

VI. *Nitrogen Determination.*—The methods we possess for the determination of nitrogen in its several forms are, on the whole, so accurate that discrepancies are easily avoided.

(1) *Nitric Nitrogen.*—A number of processes have for years been known by which the old indirect method* of analysis of nitrate of soda can be replaced by the direct determination of the nitric nitrogen. These methods depend upon the reduction of the nitric acid by nascent hydrogen, and conversion of the nitrogen into ammonia.

The first simple and reliable method was that of Ulsch: 0.5 gramme of the nitrate dissolved in 25 c.c. of water is placed in a flask with 5 grammes of reduced iron and 10 grammes of dilute sulphuric acid of 1.35 specific gravity. In the neck of the flask is hung a long pear-shaped glass bulb filled with cold water, which serves the double purpose of condensing the gases and preventing loss through spirting. The reaction commences at once in the cold, but after a little while the contents of the flask are cautiously warmed and finally raised to the boil. The reduction takes at the most ten minutes; when it is finished, the glass bulb is carefully rinsed with the wash-bottle, about 150 c.c. of water and 25 c.c. of soda solution of 1.30 specific gravity added, the ammonia distilled off into standard acid, and the excess of acid determined by titrating back with soda, potash or baryta. The whole analysis can be performed in half an hour. It is necessary to test the iron for nitrogen before use, as reduced iron frequently contains as much as 0.3 per cent.; should any be present, the amount so introduced must be deducted from the result.

Numerous modifications and improvements have been proposed which it is not necessary to enumerate. The method, however, of Devarda, which is equally simple and quite reliable, can be very strongly recommended: 10 grammes of nitrate are dissolved in 1 litre of water and 50 c.c. of the solution (= 0.5 gramme of substance) introduced into a flask of $\frac{3}{4}$ litre capacity. To this are added 80 c.c. of water, 40 c.c.

* It must here be remarked that the old indirect method for the analysis of nitrate of soda by determining the moisture and impurities, and taking the nitrate of soda by difference, is still the international trade method, and is, therefore, retained by the import houses.

of solution of soda of 1.3 specific gravity, 5 c.c. of alcohol, and 2.5 grammes of an alloy (in powder) consisting of 50 parts of copper, 45 parts of aluminium, and 5 parts of zinc. The flask is then at once connected with a condenser, and very cautiously and gently warmed to assist the reaction. It takes about 20 or 25 minutes, and as soon as the disengagement of hydrogen ceases, the ammonia is distilled off. The

alloy can be prepared by melting in a Hessian crucible 50 parts of copper, and adding first 45 parts of aluminium and then 5 parts of zinc to the molten mass. It can, however, be purchased ready prepared from T. Srpeck, 62, Stumpergasse, Vienna, VI. It is very brittle, and is easily pulverized.

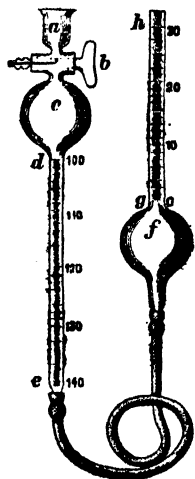
These two methods (Ulsch's and Devarda's) have taken the place of the older nitrometer method of Lunge,* though the results obtained by the latter are quite satisfactory. The following is a description of this method:

The nitrometer (see sketch) being firmly screwed in its holder, the tube *h g f* is raised, until the lower end is on a level with the three-way stopcock *b*. Mercury is then poured into the apparatus by means of a funnel, until the bulb *c* is completely filled and a little passes through the stopcock into *a*. The tap is now turned so as to shut off *c*, the tube *h g f* lowered, and about 0.360 gramme nitrate with 1 to 1½ c.c. water introduced into the cup *a*.

The solution is allowed to flow into *c*, care being taken to prevent any air from entering, and is followed by 15 c.c. of concentrated sulphuric acid (specific gravity 1.84). Gas is evolved at once, but after a time the part *a c d e* is cautiously shaken to assist the disengagement. The gas evolved forces the mercury into the bulb *f* and up the tube *h g*. The apparatus is allowed to stand for about an hour, and is then cool enough to read off. The tubes *d e* and *h g* are adjusted so that the mercury stands higher in *h g* than in *d e* by an amount corresponding to the pressure of the column of sulphuric in the latter (one division of mercury is equal to 7 divisions of sulphuric acid). The atmospheric temperature and pressure are then observed, and the volume of N_2O_2 reduced to 0° C. and 760 mm. pressure. Each c.c. of N_2O_2 corresponds to 0.003805 grammes of saltpetre.

The nitrometer can also be used for the determination of nitrogen in mixtures of nitrate and superphosphate, only in this case it is more convenient to employ the original form of nitrometer, without the bulb *c*, as so much more substance is required to produce sufficient gas to fill the bulb. If this process is used, the substance can either be weighed direct, or a portion of the solution made for the determination of the soluble phosphoric acid can be taken. In other respects, the process is carried out as above described.

(2) *Ammoniacal Nitrogen*.—20 grammes of substance are dissolved in 1 litre of water, and 50 c.c. (equal to 1 gramme), distilled with a 5 per cent. soda solution, or with thin milk of lime, until it is of the consistence of syrup. The method is carried out as follows: the 50 c.c. of solution are placed in a hard glass flask of about 600 c.c. or 700 c.c. capacity, 40 c.c. of soda solution of 1.3 specific gravity added, and the whole



* *Chem. Industrie*, 1881, p. 347.

diluted to 250 c.c. By means of a caoutchouc cork a Reitmeier's apparatus is then attached to the mouth of the flask. This apparatus consists of a glass bulb with two glass tubes blown on to it. The tube connecting the bulb with the flask, when the apparatus is attached, is short and straight, but that inside the bulb and connecting it with the condensation tube is bent upwards. By this arrangement any spray that might pass over into the receiver is caught and returned to the flask. Tincture of cochineal makes the best indicator, as it admits of sufficiently sharp readings being obtained by gaslight.

The method lately relied upon, viz., distillation with magnesia, is untrustworthy. Its introduction resulted in numerous discrepancies not before experienced, not only in the analysis of pure ammonia salts, but also of mixed manures containing nitrogen wholly in the form of sulphate of ammonia. The theory upon which the process was based, viz., that by the ordinary soda distillation, some nitrogen, existing as organic compounds of ammonia salts, was converted into ammonia, proves to be erroneous, such compounds not occurring at all. Consequently, as it has been found to give rise to differences of as much as a half per cent. in the nitrogen with sulphate of ammonia samples, it is not for the present recommended. These differences would, however, disappear if the nitrogen *soluble in water* were determined, as in the case of the phosphoric acid.

(3) *Organic Nitrogen*.—For the determination of nitrogen existing as organic matter and ammoniacal salts, the old method of combustion with soda-lime is in the first place applicable. A combustion tube of hard glass, 40 c.m. long and 10 mm. to 12 mm. wide, is closed at one end by drawing out to a point, and sufficient fine granular soda-lime introduced to occupy 4 c.m. to 5 c.m. from the end. The weighed portion, carefully mixed with soda-lime, is then passed into the tube, followed by some coarser soda-lime, leaving a few centimetres free at the mouth for the asbestos. The nitrogen bulb is then attached, and the combustion proceeded with. At first only the part containing the pure soda-lime is heated, but afterwards by degrees the whole tube, until gas ceases to come off. The point of the tube is then broken off and air sucked through the apparatus while it cools.

The method introduced by Kjeldahl (*Zeit. Anal. Chem.*, 22, 366) is, however, now generally used in place of the above. This method has been greatly modified and improved since it was first brought out, so that now indeed almost every laboratory has in use some special modification peculiar to itself. The Kjeldahl method is based upon the principle that nitrogenous compounds, on heating with concentrated sulphuric acid, in the presence of oxidizing agents, are converted into ammonia. One gramme of substance is introduced into a hard-glass flask of 300 c.c. to 400 c.c. capacity, $\frac{1}{2}$ to 1 gramme mercuric oxide, and 15 c.c. concentrated sulphuric acid added, and the mixture heated—at first gently, afterwards strongly—until completely colourless. While being heated, the flask is supported upon wire gauze in an inclined position, and its mouth is closed by a loosely fitting long glass stopper, as a precaution against loss by spirting. A flask with a long neck answers the same purpose. After cooling, the fluid is washed out into a flask, and 150 c.c. of sodium hydrate solution of 30° Bé, 25 grammes of a 10 per cent. potassium sulphide solution, and a few small pieces of zinc added, and the ammonia is then distilled off. In

presence of nitrates this method cannot be used, as nitric nitrogen is only partly converted into ammonia. If $\frac{1}{2}$ to 1 gramme of anhydrous copper sulphate is used in place of the mercuric oxide, the addition of the potassium sulphide is not required. The numerous modifications of this method are neither simpler nor more accurate than the above, and it is, therefore, unnecessary to describe them.

(4) *Total Nitrogen*.—For the determination of total nitrogen, *i.e.*, organic, ammoniacal and nitric, we have two equally good methods, as follows:

Jodlbauer's method. 1 gramme of substance is placed in a flask, and 50 c.c. of a solution of 20 grammes of phenol in 1 litre of concentrated sulphuric acid (66° Bé) added. A combination takes place in a few minutes between the nitric acid present in the substance and the phenol, producing nitrophenol. Two to 3 grammes of zinc-powder (free from nitrogen) and $\frac{1}{2}$ to 1 gramme of metallic mercury are then added, and the mixture boiled. In from a half to three quarters of an hour the whole of the nitrogen compounds are transformed into ammonia, and amidophenol takes the place of nitrophenol. It is allowed to cool, some water cautiously added, allowed to cool again, an excess of sodium hydrate solution added and the ammonia distilled off.

Kjeldahl's method combined with Ulsch's. The weighed portion is placed in a 300 c.c. flask, and sufficient water added to bring it into a pasty consistence. From 1 to 4 grammes of reduced iron, and 5 to 10 c.c. of dilute sulphuric acid (1.35 sp. gr.), according to the approximate quantity of nitric nitrogen present, are added, and the mixture heated, as directed for Ulsch's method, until the evolution of gas ceases. Half a gramme of anhydrous cupric sulphate, and 15 c.c. of an acid solution containing 200 grammes phosphoric anhydride in 1 litre sulphuric acid are then added, and the mixture heated, very carefully at first, until a clear green liquid is obtained. Finally, the ammonia is distilled off in the usual way. This method is usually preferred to Jodlbauer's on the grounds of simplicity and rapidity of execution.

The value of the sulphuric acid used for the absorption of the ammonia is found by making repeated determination by precipitation with barium chloride.

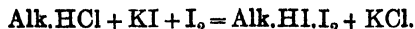
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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

TOXICOLOGICAL ANALYSIS.

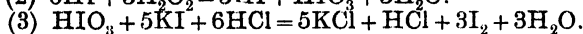
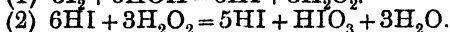
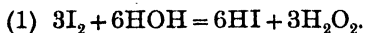
Estimation of Alkaloids by Titration with Iodine Solution. C. Kippenberger. (*Zeit. anal. Chem.*, 1896, xxxv., 10-27.)—In his former paper (*ANALYST*, Abstracts, xx., 201) the author stated that, in titrating alkaloids with a solution of iodine in potassium iodide, in most cases the free iodine combined with the alkaloid to form the compound Alk.HI.I_2 . His later investigations show that the amount of potassium iodide has considerable influence on the results. Thus, when an iodine solution containing the smallest possible amount of potassium iodide was employed, more iodine was consumed than was the case in another series of experiments in which the iodine solution contained four times as much potassium iodide. On the other hand, the amount of iodine in the precipitate varied according to the quantity

of iodine solution used. Hence, the author considers that only in certain cases is the reaction correctly described by the equation :



With strychnine the theoretical two atoms of iodine are consumed if the potassium iodide is first added, and then an excess of iodine, but with narcotine and atropine the quantity taken up is always more than two atoms.

The variations are explained by the theory that (1) the free iodine and water combine under the influence of the alkaloid superiodide; (2) that the hydriodic acid is partly oxidized to iodic acid; and that (3) iodine is again liberated and mechanically retained by the superiodide precipitate.



The fact that iodine could often be extracted from the precipitates by potassium iodide solution is urged in confirmation of this view.

Where alkaloids other than strychnine are to be estimated, it is advisable to standardize the iodine solution with known quantities of the pure alkaloid, the conditions of the actual estimation being observed as far as possible. C. A. M.

ORGANIC ANALYSIS.

On Cold Saponification, II. R. Henriques. (*Zeit. angew. Chem.*, 1896, 221-225.)—In this communication the author describes further experiments on his method of cold saponification (*ANALYST*, xxi., 67). A caution is given as to the preparation of the alcoholic soda, which must not contain any considerable amount of water, or it will not mix with the petroleum spirit. It is to be prepared by dissolving the alkali in 96 per cent. alcohol and filtering.

The process has been tried on a large number of beeswaxes, the saponification number and acid value being determined in one operation. Three grammes of the wax were dissolved in 25 c.c. of warm petroleum benzine, and immediately titrated with semi-normal alcoholic soda, phenol-phthalein being used as indicator. This gave the acid value. Twenty-five c.c. of normal alcoholic soda were then added, the whole warmed, and left for twenty-four hours in the cold, after which the excess of alkali was titrated. The ether value thus obtained, added to the acid value, gave the saponification number. The following results were obtained with five samples of pure beeswax :

	Acid Value.	Ether Value.	Saponification No.	Hübl's Ratio.
1. Beeswax, yellow	... 20.6 ...	76.5—76.8 ...	96.6—97.4 ...	3.72
2. " "	... 20.4 ...	72.9—73.9 ...	93.3—94.3 ...	3.58
3. " "	... 20.6 ...	73.6—75.1 ...	94.2—95.7 ...	3.6
4. white	... 22.4 ...	76.1—76.9 ...	98.5—99.3 ...	3.41
5. " "	... 28.4 ...	76.6—77.1 ...	105.0—105.5 ...	2.71

The high figures given by the last sample are attributed by the author to a new method which had been employed for bleaching the wax. Waxes adulterated with cerasin and paraffin wax can also be readily saponified in this way, the experiments

described proving that no saponifiable matter is left undecomposed. The acid value and saponification number of three other substances used to adulterate beeswax were:

		Acid Value.		Saponification No.	
Carnauba wax A	3.4	...	78.4
					78.4
" " B	7.0	...	83.4
					83.4
Japan wax	221.2
					220.0
Spermaceti	2.8	...	130.6
					131.4

Liver-oils gave results agreeing well with those obtained by the usual method of saponification, but with wool-fat constant figures could not be had. This is attributed to the soaps of wool-fat fatty acids decomposing in the presence of water more readily than the soaps of other acids. The substitution of alcoholic semi-normal hydrochloric acid for aqueous hydrochloric acid gave higher results, but still not always constant. Hence the author concludes that cold saponification, while applicable to all other fats and waxes, is not suitable for the class of wool-fats. Experiments on the saponification of various esters are described at length. For the most part they are readily saponified in the cold, but with acetates of the phenol series it is essential to have a considerable excess of alkali, or the results will be too low.

C. A. M.

Estimation of Caffeine by Means of Wagner's Reagent. M. Gomberg. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 331-342.)—Wagner's statement that caffeine (and one or two other alkaloids) is not precipitated by a solution of iodine in potassium iodide has hitherto been accepted without question. The author shows that this is only true of caffeine as a free alkaloid, and that when the addition of Wagner's reagent is either followed or preceded by the addition of dilute mineral acids, a dark-reddish precipitate, having the composition of $C_8H_{10}N_4O_2 \cdot HI \cdot I_4$, is obtained. When dry this periodide is a violet-blue amorphous powder, which melts at $213^\circ C$. The precipitation is quantitative, and the precipitate loses but little of its iodine when suspended in water.

The method of estimation is substantially the same as that of Kippenberger (*ANALYST*, xx., 203), the acidulated solution of caffeine being precipitated by iodine solution, and an aliquot portion of the supernatant liquid titrated with decinormal thiosulphate solution. The amount of alkaloid is calculated from the amount of iodine taken up by the formula:

$$4I : C_8H_{10}N_4O_2 :: 506 : 194,$$

i.e., one part of iodine represents 0.3834 parts of caffeine.

The test experiments were very satisfactory, the best results being obtained when the iodine was in considerable excess. In every case the caffeine solution was strongly acidulated—about 1 c.c. of strong sulphuric acid to 50 c.c. of the liquid. A large excess of acid, however, interfered with the reaction.

For the estimation of caffeine in drugs the following process is recommended:

The drug is thoroughly digested in hot water, cooled, made up to definite volume, and filtered. An aliquot portion of the filtrate is treated with lead acetate, the precipitate allowed to settle and removed by filtration. The lead in the filtrate is removed by precipitation with sulphuretted hydrogen and filtration, and the filtrate, after boiling off the sulphuretted hydrogen, is divided into two portions. To each of these a definite volume of the standard iodine solution is added, the first without the addition of any mineral acid, the second with the addition of some hydrochloric or sulphuric acid. After standing five or ten minutes, the residual iodine in the solutions is estimated as described above. Since caffeine is not precipitated in neutral solutions or in presence of tolerably strong acetic acid, any absorption of iodine in the first portion is due to other materials in the drug. This must be subtracted from the amount of iodine taken up by the second portion; the difference represents the iodine used in the formation of the periodide of caffeine. This, multiplied by 0.3834, gives the amount of caffeine in the aliquot portion of liquid taken.

The author considers that the experimental evidence on which the above method is based entirely contradicts Kippenberger's conclusions on the action of iodine solution on alkaloids (ANALYST, xx., 201, and preceding extract); and that, whatever the case may be with other alkaloids, his (Kippenberger's) theory as to the production of hydriodic acid from iodine and water does not hold good in the case of caffeine.

C. A. M.

Commercial Analysis of Glucose. V. Denamur. (*Bull. de l'Ass. belge Chim.*, 1896, ix., 341-343.)—Twenty grammes of the sample are made up to 200 grammes with distilled water, the solution filtered, and the specific gravity taken at 17.5° C. The extract corresponding to this gravity in Balling's table multiplied by 10 gives the amount of solid matter in 100 grammes of the glucose. This figure subtracted from 100 gives the moisture.

To determine the unfermentable portion, 100 grammes of the above solution are weighed into a tared Erlenmeyer flask, 1 or 2 grammes of pressed yeast added, the flask stopped with cotton-wool, well agitated, and incubated at 25° to 30° C. After fermentation the liquid is filtered, the flask washed out over the filter, and the filtrate replaced in the flask. The liquid is then evaporated to about 50 c.c. on a sand-bath, so as to completely expel the alcohol, and after cooling is again made up to 100 grammes. The specific gravity is then taken, and the corresponding figure in Balling's table multiplied by 10 gives the amount of solid non-fermentable matter in 100 grammes of glucose.

The following results were obtained with four samples of glucose syrup:

		Moisture.	Solid Matter.	Unfermentable
		Per cent.	Per cent.	Solid Matter.
				Per cent.
Syrup	(I.)	... 21.523	78.477	30.970
	(II.)	... 25.734	74.266	31.060
	(III.)	... 23.131	76.869	29.592
Very thick syrup		... 14.854	85.146	38.062

C. A. M.

INORGANIC ANALYSIS.

The Solution of Phosphates for Analysis. *Henri Lasne.* (*Ann. de Chimie Analyt.*, i., 85-89.)—The methods of dissolving the phosphate proposed by different experimenters are selected chiefly with reference to their bearing on the subsequent operations, and the final results differ in each case on account of the varying proportions of gangue attacked by the solvents.

In selecting from among them the method which fulfils the greatest number of essential conditions, attention must chiefly be directed to the solubility of the silica, iron, alumina, and especially fluorine, present, because the phosphoric acid and protoxides are sure to be dissolved whichever process be employed.

Most phosphates contain fluorides, and the hydrofluoric acid, liberated by the acid solvents, attacks the silica in the gangue, bringing it into solution as silica and fluosilicates, the presence of which is a cause of error in all the subsequent determinations. The fluorides should, therefore, be removed at the outset, and this is most conveniently effected—when volatile acids are being used—by evaporating to dryness, as in this case the fluoride of silicon is disengaged, and the silica remaining is rendered insoluble. The results will, however, differ according as the evaporation is effected in presence of the residue, or after the liquid has been separated therefrom.

As a solvent for phosphates nitric acid, used alone, is unsuitable; it attacks the ferric oxide with great difficulty, and leaves behind an unknown quantity of this substance—a circumstance sufficient to condemn it for general use.

The action of dilute sulphuric acid is very slow, owing to the slow solubility of the calcium sulphate. This acid is therefore only suitable for such special cases as when the iron is to be estimated by permanganate. Under the action of concentrated boiling sulphuric acid, as a rule nothing remains in the washed residue but silica and alumina, and, whatever acid solvent is used, a good deal of the alumina will certainly be left unattacked, and have to be determined separately in a complete analysis. Generally, however, the investigation is confined to the alumina that has become dissolved and is capable of 'reducing' the superphosphate. In practice as small an amount as possible of acid of 50° B. (specific gravity, 1.532) is used, consequently the unaffected matter is in contact with free phosphoric acid and acid phosphate of lime, and not with concentrated boiling sulphuric acid.

The varying action of the solvents on the alumina is shown in the accompanying table, the figures (which refer to the *insoluble residue*, this being the most convenient for the estimation) being given in relation to phosphorus pentoxide = 100. In the original crude phosphate the alumina amounted to 7.635 per cent. of P_2O_5 :

	Alumina.	Silica.
Residue from superphosphate	0.805	11.940
Residue from phosphate acted on by:		
1. Concentrated boiling sulphuric acid ...	0.644	7.000
2. Hydrochloric acid, boiled for ten minutes	1.152	6.222
3. Aqua regia	1.065	7.910
4. Hydrochloric acid, evaporated to dryness	0.895	10.250
5. Aqua regia	0.807	10.200

This shows that the action of boiling concentrated sulphuric acid is too energetic, and that of the volatile acids is the reverse, the results obtained in practice being most nearly approached by employing the latter solvents and evaporating to dryness without removing the residue. In order to render the silica completely insoluble the residue must be moistened and re-dried several times. It is then taken up with a minimum quantity—say 1 c.c. of 20° B. (specific gravity, 1.161) hydrochloric acid, and 20 c.c. of water per gramme of phosphate—maintained at a temperature of about 100° for at least half an hour, and filtered through a tared filter into a graduated glass, the liquid being afterwards made up to 100 c.c. per 1 gramme of phosphate; or the liquid may be made up to the desired volume before filtering.

The addition of nitric acid to the hydrochloric acid will destroy most, if not all, of the organic matter frequently present in crude phosphate, but it is perhaps better to use the latter acid alone, and to estimate approximately both the organic matter left in the residue and the small quantity that passes into solution.

The evaporation is generally performed in a flat porcelain capsule over a sand-bath, avoiding heating the latter too strongly, but where the residue is small, or greater accuracy is required, a platinum dish must be used, in which case no nitric acid should be added. It is sometimes useful to add a known quantity of silica, which is subsequently deducted from the weight of the residue. When higher oxides of manganese are present their action may be neutralized by a little oxalic or formic acid, or alcohol. If oxalic acid be used any excess in the solution must be neutralized.

The estimations are made with aliquot parts of the hydrochloric acid solution, portions of which may, if desired, be transformed into nitrates or sulphates by evaporation in presence of an excess of the corresponding acids.

C. S.

Inaccuracies in the Estimation of Manganese in Ferro-Manganese. H. von Jüptner. (*Oesterr. Zeits. Berg. u. Hüttenw.*, 1896, xliv, 15; through *Chem. Zeit. Rep.*, 1896, 52.)—Although one cause of the want of agreement often shown in these analyses is due to the different atomic weights used in the calculations, the chief reason lies in the errors of standardizing the permanganate employed. Mohr's salt, ferrous ammonium sulphate with 6 molecules of water, is apt to contain ferric oxide, and it is also liable to lose ammonia in the presence of stronger bases. On exposure to air, it changes to a ferric ammonium sulphate with (theoretically) 3 molecules of water; but as more than this amount is given off during the action of the atmospheric oxygen, the crystals are liable to be damp. The use of steel of known composition is to be avoided, as some of the metal may become oxidized during solution in the acid, while the combined carbon is capable of reducing permanganate. Oxalic acid gives exact results and is the best substance to employ.

F. H. L.

THE ANALYST.

AUGUST, 1896.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

SUMMER MEETING.

THIS took place on the afternoon of July 14, when about thirty members and their friends lunched together, and proceeded by steam-launch to the sewage outfall works at Barking, where a tour of inspection was made, under the guidance of Mr. W. J. Dibdin. The party afterwards returned to Greenwich, and dined at the Ship.

THE ANALYSIS OF MORTAR.

By W. J. DIBDIN, F.I.C., AND R. GRIMWOOD, F.I.C.

(Read at the Meeting, June 3, 1896.)

The Legal Specification of Mortar.

In the by-laws made by the London County Council, under Sect. 16 of the Metrop. Management and Building Acts Amendment Act, 1878, it is provided that the mortar used under that Act must be composed of freshly-burnt lime and clean sharp sand or grit, without earthy matter, in the proportions of one of lime to three of sand or grit.

In the case of cement-mortar, the cement to be used must be Portland cement, or other cement of equal quality to be approved by the district surveyor, mixed with clean sharp sand or grit, in the proportions of one of cement to four of sand or grit; also that burnt ballast or broken brick may be substituted for sand or grit, provided such material be properly mixed with lime in a mortar-mill.

In the by-laws made by the Council under Sect. 31 of the London County Council (General Powers) Act, 1890, it is provided that plastering or coarse stuff shall be composed of lime and sand, in the proportion of one of lime to three of sand, mixed with water and hair; but Portland, Keene's, Parian, Selenitic, or other cement or plaster of Paris, may also be used for plastering. The lime must be freshly-burnt lime; the sand must be clean sharp sand, free from loam or earthy matter; the hair must be good and sound, free from grease or dirt, and that one pound of hair to be used to every three cubic feet of coarse stuff. Fibrous material may be used instead of hair, and ground brick or furnace slag, each to the satisfaction of the district surveyor, may be used instead of sand; and the setting coat must be composed of lime or cement mixed with clean washed sand, or cement only.

Usual Character of Mortars.

Earthy Matter.

From these extracts from the by-laws relating to the use of building materials in the Metropolis, it will be seen that an analysis of lime-mortar or cement-mortar,

etc., can be conducted with a degree of accuracy sufficient to indicate very slight departures from the prescribed quantities of the materials used. Unfortunately analyses which have been made from time to time by us have shown distinctly that it is only under exceptional circumstances that mortars which come within these regulations are employed. To take one factor only, the earthy matter: analysis of samples of mortar, made with materials selected as coming within the definition of the by-laws, and in the proportions therein set out, shows that the earthy matter, as determined by us in the manner described hereafter, varied as follows: 1.5, 2.1, 0.14, 0.74, 0.81, 0.61, 0.64, 0.96, 0.85, and 0.70 per cent.; thus showing that the finely-divided matters which may be accidentally introduced by means of the broken brick or sand form but a very small proportion of the actual weight of the mortar. As compared with these, the following results obtained from the examination of a number of samples of mortars taken from a building in course of erection will indicate the great difference in this respect, viz.: 4.9, 3.8, 9.1, 3.4, 14.7, 13.7, 7.4, 10.4, 8.1, 12.3, per cent.

Soluble Silica.

Another factor of importance in estimating the quantity of cement in a genuine cement-mortar is that of the soluble silica. As is well known, the silica, soluble in hydrochloric acid, present in Portland cement will vary between 17 and 20 per cent.; but if the average is taken at about 18 per cent., it will form a fair basis for calculation.

If the cement-mortar is made with good Portland cement and clean, well-burnt brick or sand in the proportion of one of cement to four of the grit, there will be about $8\frac{1}{2}$ per cent. of soluble silica. In some samples of genuine cement-mortar, the soluble silica was found to be 3.25, 2.5, 3.25, and 2.25 per cent.; as against these samples of *genuine* mortar made with lime gave soluble silica equal to 0.9, 1.0, 0.6 traces, and 1.1. If we now turn to the analyses of *commercial* mortars (see Table V.) above referred to, we find that the soluble silica varied as follows: 0.9, 0.7, 0.7; thus showing a very marked difference between this factor under different circumstances.

Carbonic Acid.

A third factor is the quantity of carbonic acid. Genuine mortars made with good materials were found to contain, after the mortar had been made for some three months, carbonic acid equal to 1.02, 1.36, and 1.06 per cent. of carbonate of lime; and the cement-mortars to contain carbonic acid equal to 2.86, 2.86, 1.04, 1.87, 1.45 per cent. of carbonate of lime. Freshly prepared mortar made with good materials contained in two instances 0.3 and 0.4 per cent. carbonate of lime. Against these, the commercial mortars contained 6.42, 6.24, 7.39, 4.52, 8.9, 8.4, 8.0, and 4.5 per cent.; thus showing conclusively that this factor alone gave such marked indications of the character of the material that it would be almost impossible that a mistake should be made in estimating the character of a given sample of mortar or cement. It is to be noted that certain bricks contain carbonate of lime, and this should be determined before concluding that the mortar is made with old materials or insufficiently burnt limestone. In Table I. an instance is given of a brick containing 7.77 per cent. of carbonate of lime. The full details of these analyses are given in the tables at the end of the paper.

Relation of Weight to Volume (Lime-Mortar).

In the by-laws it is provided that the quantities are to be by volume, and not by weight; therefore the analysis, being stated by weight, must be converted into terms of volume. In order to show how this is arrived at, the following comparative analysis of a sample of mortar, specially prepared for this purpose, will make the point clear. The analyses of the lime and broken brick are given in Table I.; and, in Table II., that of the mortar made with one volume of lime and three volumes of brick. In this case the brick had some old mortar adhering to it, as would happen where old bricks are used.

TABLE I.

Sample of the Lime used in making Mortar.

Lime (CaO)	78.40 per cent.
„ carbonate	3.22 „
„ sulphate	0.64 „
Soluble silica, iron oxide and alumina	12.85 „
Sand	4.80 „
Total	99.91 „

Sample of the Brick used in making Mortar.

	Per cent.
Moisture (loss at 212° Fah.)	1.27
Lime (CaO)	1.99
„ carbonate	7.77
„ sulphate	1.03
Soluble silica, iron oxide and alumina	3.60
Insoluble matter (crushed brick)	78.77
Insoluble matter (fine)	1.69 { This had the appearance of having been burnt.
Total	96.12

TABLE II.

Sample of Mortar made with One Part by Measure of Lime and Three Parts Crushed Brick.

	Per cent.
Moisture, water of hydration, etc.	29.71
Lime (CaO)	12.85 = 16.06 commercial lime.
„ carbonate	4.47
„ sulphate	0.61
Crushed brick	46.03
Iron oxide and alumina	2.80
Soluble silica	1.85
Earthy matter	0.70 { This had the appearance of having been burnt.
Loss on ignition	0.98
Total	100.00

Commercial lime (containing 80 per cent. of CaO) to crushed brick, 1 to 2.29
by volume.

„ „ „ „ „ to all other matters, 1 to 2.86
by volume.

In this it will be seen that the earthy matter was 0·70 per cent. in the mortar, and the lime (as pure lime) 12·85 per cent., equal to 16·06 per cent. commercial lime of 80 per cent. pure CaO.

The brick (Table I.), as already mentioned, contained 7·77 per cent. of carbonate of lime. Another instance is given in Table III. of the analyses of lime and mortar made therewith and good brick. In this the carbonate of lime was only 1·97 per cent. on the mortar.

TABLE III.

Analyses of Stone-lime and Mortar made therefrom :

	Stone Lime.	Mortar. Lime 1 : Brick 3 parts by Volume.
Moisture, water of hydration, and loss ...	1·86	31·23
Lime (CaO)	78·63	10·89
Carb. lime	0·93	1·97
Sulphate of lime	None	0·61
Iron oxide and alumina	4·30	3·55
Soluble silica	7·10	1·11
Sand and grit	Trace	45·99
Earthy matter	6·20	1·37
Loss on ignition	0·98	3·28
	100·00	100·00

A large number of experiments on different samples of ground-up brick and various samples of lime, etc., have shown that it is a safe guide, in determining the volume of the materials from their weight, to increase the weight of lime found in the analysis by one-fourth in order to raise the lime (CaO) obtained to the relative quantity of *commercial* lime, on the assumption that the commercial lime originally contained 80 per cent. of pure oxide of calcium. The correction from weight to volume is then made by again increasing the weight by one-fourth.

The following will illustrate the method :

Equal volumes of stone-lime and of air-dry broken brick weighed as follows :

Lime	17·38 grammes per 18·5 c.c.
Brick	21·13 " " "

(1) To convert weight of commercial lime to equal volume of broken brick, add one quarter of the weight of lime found to itself, thus : $17·38 + 4·34 = 21·72$.

(2) By analysis, this lime contained 78·63 per cent. of real CaO, and a mortar made with this lime on analysis gave 10·89 per cent. of CaO ; therefore, to convert CaO in mortar into terms of commercial lime, for practical purposes add one quarter of the weight of CaO found to that quantity. Thus, CaO in mortar 10·89 per cent. $+ 2·72 = 13·61$ commercial lime.

(3) By No. 1, $13·61 + 3·4 = 17·0$ of CaO by volume.

(4) The ground-brick used in making the mortar (Table III.) contained 6 per cent. of matters soluble in weak acid ; therefore, to the weight of brick-grit actually found add 6 per cent., *i.e.*, sand and grit (brick), $45·99 (45·99 \times 0·06 = 2·76) = 48·75$.

(5) Lime to brick by volume : 17 : 49, or 1 : 3 nearly.

Relation of Weight to Volume (Cement-Mortar).

Equal volumes of washed and dried sand and of Portland cement of specific gravity 3.15 weighed 21.7 grammes and 19.3 grammes respectively. Therefore, the weight of cement found must be raised by one-eighth to equal the sand by volume.

Average Portland cement will contain from 17 to 20 per cent. of soluble silica. An average of 18 per cent. may therefore be taken, on which assumption the soluble silica formed may be calculated into terms of cement. Thus the 3.25 per cent. of soluble silica found in Sample No. 1, Table IV., would equal 18 per cent., or 19.25 per cent. corrected for volume. The sand and grit equalled 67.34 per cent.; therefore the ratio of cement to sand was 1 : 3.5. In sample No. 2 of the same table the soluble silica was $2.5 = 13.9$ per cent. of cement = 15.6 by volume; the sand, etc., was 70.77 per cent.; the ratio consequently was 1 : 4.5. Sample No. 3 showed a ratio of 1 : 3.5, No. 4 of 1 : 5.6, and No. 5 of 1 : 4.8, the average being 1 : 4.4. The samples were stated to be from cement-mortars, made with 1 of cement to 4 of sand. Doubtless the difference is due to unequal mixing.

Absorption of CO₂ after Mortar is made.

The crushed brick (see Table I.), including some of the silica, iron oxide, and alumina, would amount to 48 per cent., or 51.77 per cent., including 3.74 per cent. of carbonate of lime found on analysis to be present in the brick. The commercial lime used contained 3.22 per cent. of carbonate of lime, so that the above 16.06 per cent. would contain 0.52 per cent. These together will account for 4.26 per cent. of the carbonate of lime in the quantity found in the above analysis, showing that only 0.21 per cent. of carbonate of lime was formed between the times when the mortar was made and analysed.

Analytical Methods.

Various suggestions have been made as to the best means of more or less rapidly arriving at a conclusion as to the quality of a mortar. Our own experience is against the adoption of any short cuts. However strong one's individual opinion may be, the evidence is incomplete unless the work is thoroughly done. It may be interesting here to shortly recite the outlines of the method adopted by us in these examinations.

First, the sample, having been thoroughly well broken up and mixed, is placed in a suitable bottle, from which 10 grammes are weighed in a platinum dish and placed on the water-bath for the estimation of moisture, the drying being continued in the usual way until no further loss is found. Objections may be made to the adoption of this system, in so far as it may be assumed that carbonic acid will be absorbed by the mortar during the process of drying; but a large number of experiments have shown that such absorption, if any, makes a difference so little in degree that it obviously falls within the limits of the allowable quantity in good samples of mortar. A further portion, weighing 10 grammes, is placed in a beaker, stirred up with 10 per cent. hydrochloric acid, and allowed to stand for one minute, when the fluid is decanted, and with it all the fine earthy matter held in suspension. This process is repeated until the supernatant water at the end of one minute is clear. The cleaned, washed sand and grit or broken brick is then dried and the weight ascertained. The earthy matter is filtered from the washings and

weighed; a known portion of the filtrate is taken for the estimation of the silica, iron, alumina, and lime, the latter being weighed as sulphate.

The sample taken for moisture after weighing is ignited at a dull red heat, insufficient to decompose the carbonate, and the loss on ignition so found recorded.

The ignited mortar is then treated with a small quantity of water to completely hydrate the lime, dried at 220° Fahr. and weighed. The loss on ignition is thus obtained. If found necessary, the carbonic acid may be again taken on the dried mortar as a control.

It is obvious that the analysis may be more or less complete according to the character of the information required.

The appended tables, Nos. IV. to VII., contain the results of the analyses of some typical samples analysed by us.

Table No. IV. shows the results of the analyses of five samples of cement mortar.

Table No. V. shows the results of the analyses of three samples of bad mortar.

Table No. VI. shows the results of partial analyses of fifteen samples of very bad mortar; in these the analyses were carried only far enough to show that all the samples contain excessive quantities of earthy matter and old mortar; and also that they were deficient in freshly burnt lime.

In contrast with these, Table No. VII. shows the result of the partial analyses of a sample of good mortar taken from a public building in course of erection. In all the following tables the results are stated in percentages.

TABLE IV.

Analyses of Samples of Cement-mortar.

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Moisture, water of hydration, etc.	7.50	6.12	7.72	7.26	9.68
Lime (CaO)	11.80	10.87	11.97	9.20	11.24
„ carbonate	2.86	2.86	1.04	1.87	1.45
„ sulphate	0.20	0.30	0.99	0.84	0.71
Iron oxide and alumina	4.35	3.75	4.00	4.95	4.00
Soluble silica	3.25	2.50	3.25	2.00	2.25
Earthy matter	0.61	0.64	0.96	0.85	0.70
Loss of ignition	2.09	2.19	2.42	2.50	2.02
Sand and grit	67.34	70.77	67.65	70.63	67.95
Total	100.00	100.00	100.00	100.00	100.00

Ratio of Cement to Sand, etc.

Calculated on the Soluble Silica.

1 : 3.5 ... 1 : 4.5 ... 1 : 3.5 ... 1 : 5.6 ... 1 : 4.8

Calculated on the Lime.

1 : 2.7 ... 1 : 3.0 ... 1 : 2.9 ... 1 : 3.0 ... 1 : 3.0

The average total lime in cement may be taken as 60 per cent.; therefore, the lime found, including that carbonated, may be corrected to volume of cement thus :

Sample No. 1— $\text{CaO} = 11.8 + 1.6 \text{ carbonated} = 13.4 = 22.3$ of cement; add one-eighth for correction to volume = 25.1 vols. of cement to 67.34 of sand, or a ratio of 1:2.7.

It will be noticed, however, that the calculation of cement from the lime found is unsafe, as the mortar might be sophisticated with lime, and therefore not *cement-mortar*. The soluble silica is the only reliable indicator.

TABLE V.
Bad Lime-mortars.

	No. 1.	No. 2.	No. 3.
Moisture, water of hydration	17.1	21.1	15.1
Lime (CaO)	5.9	6.0	6.2
„ carbonate	8.9	8.4	8.0
„ sulphate	1.8	1.7	2.0
Sand, grit, broken brick, etc.	51.5	46.3	53.4
Iron oxide and alumina	1.6	1.4	1.5
Soluble silica	0.9	0.7	0.7
Earthy matter	7.4	10.4	8.1
Loss on ignition	4.9	4.0	5.0
Total	100.0	100.0	100.0
Commercial lime (CaO) to sand and grit, etc., by volume	1 to 5.5	1 to 4.9	1 to 5.5
„ „ to all other matters by volume	1 to 8.3	1 to 7.7	1 to 8.1

TABLE VI.
Partial Analyses of very Inferior Mortars.

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.	No. 7.
Moisture	3.13	3.61	3.42	2.80	2.79	3.00	3.22
Lime (CaO)	7.48	8.72	5.68	7.71	6.11	6.00	6.73
Lime carbonate	12.61	13.18	15.45	11.36	15.79	14.66	16.34
Sand, grit, etc.	58.90	54.85	57.14	58.76	59.09	55.36	55.84
Earthy matter	8.44	7.39	8.68	7.57	7.65	9.94	9.27
Iron oxide, alumina, and soluble silica	3.75	2.90	3.00	4.50	3.30	2.60	3.20
Commercial lime (CaO) of 80% to all other matters of volume	1 to 5.0	1 to 4.2	1 to 6.4	1 to 4.8	1 to 6.1	1 to 5.9	1 to 5.3
Commercial lime (CaO) of all other matters by volume	1 to 7.6	1 to 6.4	1 to 10.2	1 to 7.4	1 to 9.5	1 to 9.7	1 to 8.5

	No. 8.	No. 9.	No. 10.	No. 11.	No. 12.	No. 13.	No. 14.	No. 15.
Moisture	3.87	10.28	4.64	3.68	3.98	7.97	3.15	6.32
Lime (CaO)	9.97	9.00	7.50	12.01	7.63	10.29	8.34	5.96
Lime carbonate	11.25	11.13	15.11	8.86	15.80	8.63	16.70	20.95
Sand, grit, etc.	57.00	50.96	50.35	55.27	50.60	54.27	50.35	52.05
Earthy matter	8.10	8.78	9.30	8.63	8.78	8.70	10.41	8.23
Iron oxide, alumina, and soluble silica	4.50	3.60	3.80	4.15	4.70	3.70	4.20	3.30
Commercial lime (CaO) of 80% to sand, grit, broken brick, etc., by volume	1 to 3.6	1 to 3.6	1 to 3.9	1 to 2.9	1 to 4.1	1 to 3.8	1 to 3.8	1 to 5.5
Commercial lime (CaO) of 80% of all other matters by volume	1 to 5.5	1 to 5.6	1 to 6.9	1 to 4.4	1 to 7.2	1 to 5.0	1 to 6.7	1 to 9.4

TABLE VII.

Sample of Good Mortar taken from a Public Building in Course of Erection.

Moisture	17.84
Lime (CaO)	14.43
Lime carbonate	0.97
Sand, grit, etc.	59.15
Earthy matter	1.05
Iron oxide, alumina, and soluble silica...	3.75

Commercial lime (of 80 per cent. CaO) to sand, grit, etc., by volume, 1 to 2.6.

„ „ to all other matters (dry), 1 to 3.0.

DISCUSSION.

The PRESIDENT said that it was seldom the Society had the pleasure of Sir Charles A. Cameron's presence at their meetings, and he was sure he only echoed the sentiment of all members present in asking Sir Charles to open the discussion.

Sir CHARLES A. CAMERON said he was afraid he could not add anything to the very valuable information that had been laid before the Society by the authors of this paper. He occasionally had specimens of cement submitted to him which, although apparently having a correct composition—according to what was laid down in the text-books—nevertheless would not adhere to the surface of bricks. As a matter of fact, disputes were constantly arising in connection with cement, mortar, and concrete, and there was a very large field open to workers in the direction of obtaining information which would enable such disputes to be settled on a scientific basis.

In Ireland he did not think it was customary to use ground bricks in place of sand to such an extent as appeared to be the case in England.

He believed the authors had mentioned the case of a mortar in which the proportion of lime to sand was as low as 1 : 9. He had himself once examined a sample containing almost as much sand, viz., 86 per cent.

Mr. ALLEN said that one problem which he had been called upon to solve was how much road-sweepings a particular sample of mortar contained, and what was the proportion of "clean, sharp sand." Road-sweepings seemed to be a very indefinite article, and he thought that if the authors could indicate how to ascertain the quantity present, it would be of great service to others as well as to himself.

It seemed to him, if he had understood that part of the paper correctly, that it was a little questionable whether it was fair to deduce the amount of cement in a cement mortar from the amount of soluble silica. This surely must be a very variable quantity.

There was no doubt that the Society was very much indebted to anyone bringing forward figures on such a difficult subject, as they would at least afford something like a basis for further investigations.

Mr. BODMER remarked that where broken bricks were used in substitution for sand, the by-laws, although providing for the proper grinding up of the bricks, did not specify distinctly that they should be well burnt. Badly-burnt bricks might be

used, and in that case the amount of earthy matter present would be considerable. He thought it desirable that either the use of bricks should be altogether prohibited, or that some more definite limit should be laid down as to their employment.

The proportion of quicklime contained in the lime used was also a matter upon which disputes often arose, but it was hardly possible to suggest any definite standard, for in some kinds of lime which were really very good for mortar-making the percentage of actual quicklime was comparatively low.

Mr. JOHN HUGHES said that the by-laws of the London County Council in regard to the composition of mortar were far too vague. Nothing is mentioned respecting the quality of the lime, except that it should be "freshly burnt," its richness in CaO and soluble silica is entirely omitted, as well as its weight per cubic foot, which may vary considerably. The sand must be sharp and clean, "without earthy matter," but there is no definition of what constitutes earthy matter.

In making mortar it is usual to specify quantities by measure rather than by weight, consequently the respective amounts of lime and sand, or substitutes for sand, must vary in proportion to their gravity. Therefore mortar, though made according to the strict reading of the by-laws, may contain as little as 10 parts of lime (CaO) by weight in every 100 parts of the perfectly dry mortar. Thus, taking a cubic foot of gray lime containing 80 per cent. CaO , and weighing (according to Hurst's "Architectural Hand-book") 44 lbs., and 3 cubic feet of clean sand weighing 100 lbs. per cubic foot, we have a mixture in the dry state which contains only 10.23 CaO in every 100 parts by weight. On the other hand, if a substitute for sand, such as broken brick, having a less gravity, be used, the percentage of CaO in the mortar would be increased.

Some years ago he devoted considerable time to the examination of old mortar obtained from numerous old abbeys and castles, and the results of the analyses were published in *The Builder* during the years 1892 and 1893. These analyses corroborated Mr. Bodmer's observation, that the percentage of actual quicklime was really no criterion of the quality of the mortar. The lime rapidly becomes converted into carbonate, especially in the smoky atmosphere of towns, and in the case of good building-lime the CaO is associated with soluble silica in the same form as it exists in Portland cement, so that the total lime in its various combinations, rather than the caustic lime alone, should be considered before condemning a mortar.

He would mention that, according to his researches, the average amount of caustic lime in ancient mortar did not exceed 0.5 per cent. Further, within certain limits the actual percentage of total lime was no reliable indication of the quality of the mortar. Thus, the mortar from Rochester Castle (of which only the keep remains), contains as much as 28.67 CaO , while that from Tintern Abbey, in Monmouthshire (which was remarkable from the fact that the four gable ends were still standing), contains only 18.84 CaO per cent. The mortar from the leaning tower of Caerphilly Castle, in Glamorganshire, contains as little as 13.49, associated, however, with 9.85 of soluble silica.

He could not agree with Mr. Dibdin in calculating the probable amount of cement present in a mortar from the figures for soluble silica. Limestones contain varying quantities of gelatinous or hydrated silica, which after calcination forms a natural

cement, and it is to the presence of this cement that the durability of ancient mortar largely depends.

Greystone lime contains 9 per cent. of this soluble silica; Aberthaw lime contains as much as 15, while the best Portland cement contains not more than from 20 to 22; consequently limestones are suitable for making mortar in proportion to their richness in this so-called soluble silica.

In conclusion he would say that some of the best and most durable of these old mortars contained as much as 4 to 5 per cent. of oxide of iron and alumina.

Mr. B. E. R. NEWLANDS said he presumed this paper was intended to refer only to cases occurring in those districts in which the by-laws referred to had been adopted; but there were districts where the by-laws had not been adopted, and a still greater number where they had not been enforced.

In Manchester, and other large towns, mortar was manufactured as an industry by the Corporation, and this mortar was generally made with the cinders and clinkers from the Corporation's destructors. This mortar—which was sold to the general public by the Authorities—would yield very different analytical results from the mortars dealt with in the paper.

Mortar made with furnace clinker was also used to a considerable extent in the Metropolitan area, most frequently, perhaps, in the case of buildings erected by work's proprietors, whose furnaces supplied the clinker; and this mortar, although of a very high standard as far as hardness and setting power were concerned, would not satisfy the tests given in the paper.

Dr. M. A. ADAMS said, that from the local experience he had gained in the midst of the cement district, where there was an abundance of materials for making lime from both white and gray chalk, he could confirm the remark made by Mr. Hughes, that alumina was often a somewhat important constituent of commercial lime.

It was customary in his neighbourhood to look upon the lime made from gray chalk (which contained a large proportion of silicates and 3 or 4 per cent. of alumina) as almost equal in quality to Portland cement. Certainly, it was a fact well-known to all practical builders in the district, that they could use far less of such lime than was required if they employed lime made from the upper layers of the chalk, which was nearly pure calcium carbonate.

Mr. DIBDIN said that he and his colleague congratulated themselves on having raised such an interesting discussion. He sincerely hoped that the paper would be the forerunner of several others on the same and kindred subjects.

Mr. Newlands was perfectly correct in his remarks as to the scope of the paper. The question had been dealt with entirely as under the by-laws relating to London. As a matter of fact, these by-laws contained the only statement or definition of mortar having legal weight of which he was aware. If they were wrong, it was without doubt desirable that they should be amended as soon as possible, but as they existed, a considerable amount of work had been done under them, and it was just as well that they should be thoroughly understood.

The subject, taken in its industrial aspect and as ranging over the ground of all kinds of mortar, both ancient and modern, was of course a very large one, and to treat it exhaustively would require a great deal more than the hour or so available at one

of the Society's meetings, and he and his colleague looked upon this paper rather as an index for pointing out that a large amount of valuable work could be done by those who took up the subject in detail.

With regard to the adulteration of mortar, it was seldom that a sample was found which really complied with the specification contained in the by-laws, a common adulterant, in London at any rate, being dust-bin refuse.

Mr. Allen had asked for information as to the best mode of estimating road-sweepings. In many cases road-sweepings consisted largely of sharp sand mixed with organic matter. The only way to deal with them was to separate the organic from the mineral matter and estimate them in the ordinary way. Of course quite a cursory observation would indicate whether the organic matter was in the form of horse-dung or the like.

With regard to Mr. Bodmer's remarks, he did not think much difficulty would be found in differentiating between properly burnt earthy matter and clay that had merely been warmed up. The by-laws appeared to be perfectly definite in their references to the ballast or bricks that might be used in making mortar. They stated that burnt ballast or broken brick might be substituted for sand or grit. Now, earthy matter that had merely been warmed could not properly be described as "burnt ballast," and unless a brick had been fired—and properly fired—it could not, according to his (Mr. Dibdin's) idea, be regarded as coming within the meaning of the term "brick," any more than partially heated chalk could be regarded as lime.

Mr. Hughes had remarked that the amount of lime present was no criterion of the quality of a mortar, and had referred to the mortar of Tintern Abbey as containing 16 per cent. of lime. Now, assuming 16 per cent. of caustic lime to be equivalent to 20 per cent. of commercial lime, and adding one-fourth to correct for the difference in specific gravity between the lime and sand, the volumetric proportion of lime to sand worked out in this case to 1:3, or exactly the same as laid down in the by-laws.

With regard to clinker-mortars, these were not comprised in the present paper, which dealt only with good brick, grit, or clean, sharp sand, and lime or Portland cement. In the case of mortar made with other materials, other data must be obtained as a basis of calculation.

NOTE ON "DRAWN" OR EXHAUSTED CARAWAYS.

BY BERNARD DYER, DSC., AND J. F. H. GILBARD.

(Read at the Meeting, May 6, 1896.)

ATTENTION was recently directed to the adulteration of caraway "seeds" by the admixture of "drawn" or exhausted caraways (*i.e.*, "seeds" from which the flavouring matter had been extracted). We obtained a specimen of "drawn" caraways and examined it, together with genuine caraways of good quality.

The exhausted sample was much darker in colour, and had lost the pleasant aromatic smell of unexhausted "seeds."

The following are the results :

	Loss at 100° C.	Approximate Volatile Essential Oil.	Fixed Ether Extract.	Alcohol Extract after Ether Extract.	Ash less Sand.	Ash Soluble in Water.	Ash Insoluble in Water.	Silica.
Genuine Dutch caraways .	12.3	1.9	20.4	11.6	5.8	2.1	3.7	0.3
" " " " " "	11.3	1.5	19.5	9.5	6.3	2.1	4.2	0.4
" Drawn " caraways " ...	6.9	0.1	16.1	12.0	6.3	2.2	4.1	0.4

The "approximate volatile oil" was obtained in the same manner as described by us in a previous paper on ginger (ANALYST, xviii., 197), viz., by taking the flask containing the ether extract and drying it on a shelf over the water-oven until it loses only a milligramme or so in ten minutes. It is then dried to constancy inside the water-oven to furnish the "fixed ether extract," and the difference is called "volatile essential oil."

From the above figures it will be noticed that the effect of exhausting the caraways has been to cause almost the total disappearance of the volatile oil upon which the aroma of the seeds depends, while the "fixed ether extract" has decreased by one-fifth. The other items, with the exception of the moisture, remain practically the same.

In our opinion, therefore, samples showing a low percentage of "volatile essential oil" and "fixed ether extract," and containing, if unground, many dark seeds, should be viewed with suspicion.

LEAD IN A SAMPLE OF CANADIAN CHEESE.

By F. W. STODDART, F.I.C.

(Read at the Meeting, May 6, 1896.)

THIS specimen of cheese was of a brand largely imported into England from Canada, and which in this particular instance was complained of by the importers as being very dark in the interior. On examination, it was found that the coloration, which to the eye exactly resembled moulding, was due, not to the presence of fungi, as might at first have been supposed, but to an amorphous powder, which consisted of lead dust. The specimen examined contained an average quantity of $1\frac{1}{4}$ grains of metallic lead per lb. of cheese, distributed in veins, and giving the cheese a marbled appearance.

My first supposition was that the lead had been added with the object of producing that blue appearance which was generally admired in cheese, but I was

informed by the shippers that this particular cheese was sold as a white cheese, and that the coloration would materially depreciate its value.

Regarding the form in which the addition was made, I could only conclude that the lead had been added in the form of actual lead dust. It occurred to me that it might have been added in the form of lead sulphide, but I was unable to detect any traces of sulphides, and could not imagine any chemical change which would result in the reduction of the sulphide to the metallic form actually present in the cheese. It was, however, very difficult to get the lead out sufficiently pure to make quite certain, on account of its intimate incorporation with the fatty material of the cheese.

NOTE ON THE ANALYSIS OF CREAM OF TARTAR.

BY ALFRED H. ALLEN.

A SOMEWHAT curious error occurs in my proposed process for the analysis of cream of tartar published in the July number of THE ANALYST. The facts are correctly stated in the earlier part of the paper, but in method 2 *a*, on page 180, mistakes occur which would be liable to cause confusion if left uncorrected. The following is an amended description of the process in question:—

Titrate the filtrate with decinormal hydrochloric acid and methyl-orange. In a pure sample the measure of acid required will be exactly equal to that of the alkali consumed in process 1. The presence of calcium tartrate in the sample does not affect the results. Each c.c. of *deficiency* of acid represents 0.36 per cent. of calcium sulphate (CaSO_4), or 0.72 per cent. of acid potassium sulphate (KHSO_4). Any *excess* of acid required points to the presence of neutral potassium tartrate, each c.c. of difference representing 0.60 per cent. of that salt. If the titrated liquid be treated with barium chloride, the weight of the precipitate of barium sulphate will give the means of directly determining the proportion of calcium or potassium sulphate.

The use of decinormal alkali for the titration of the original sample of cream of tartar involves the use of what may be considered an inconveniently large volume of the standard solution. If seminormal alkali be employed instead of decinormal, and 3.762 grammes be taken for analysis, each c.c. used for neutralization will represent 2.5 per cent. of acid potassium tartrate. In this case the titration will require much care, but the results will be quite accurate enough for most purposes.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD ANALYSIS.

The Colouring Matter of Natural Waters. Ellen Richards and J. W. Ellms. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 68-81.)—The estimation of the relative depth of colour in natural waters is often of considerable importance, especially in places where the water is of surface origin. In the colouring matter there are probably several complex substances—tannins, glucosides, etc., and in some cases iron. While

it is possible that nitrogen is often an essential constituent, yet experiments show that a large diminution in the nitrogen does not necessarily produce a corresponding lessening of the colour. Humic acid is also probably one of the compounds.

One of the earliest attempts to fix a standard of comparison so as to limit the allowable amount of colour was by oxidation with potassium permanganate. In a general way a rise of colour is followed by a rise in the oxygen consumed, but the results are often misleading.

The Nesslerized ammonia standards of Professor Leeds (*Proc. Am. Chem. Soc.*, ii., 8) have been largely adopted, but are open to the objection that the tints are not of absolute quantitative value, but vary according to the different methods of preparing the Nessler solution. The metallic standard of platinum and cobalt (Hazen, *Amer. Chem. Jour.*, xiv., 300) is satisfactory where only low colours are dealt with, but the authors find that the higher portions of the scale in a wide range of colour cannot be matched in this way with any degree of accuracy. They consider that the most reliable method is the use of stock bottles of the natural waters commonly met with. But since these, however carefully prepared, fade in time, some system of standardization is necessary, and formerly Professor Leeds' standard was taken as the basis. The natural waters were diluted with distilled water, so as to correspond with the Nesslerized ammonia standards at a number of selected points. They now strongly recommend the use of Lovibond's tintometer for this purpose, and give tables showing the differences in the tintometric readings of red and yellow for each tenth of colour in the natural water standard scale.

C. A. M.

Estimation of Alcohol in Wine by the Ebullioscope and Distillation. J. Sinibaldi. (*Ann. de Chimie Analyt.*, i., 126.)—The ebullioscope, which is generally employed in commercial determinations of the alcoholic content of wine, is, if properly adjusted, capable of giving accurate results, not exceeding those obtained from a carefully conducted distillation, by more than 0.1 to 0.2 gramme per litre in the case of wines containing up to 25 grammes of dry extract per litre, and approximating still more closely where the extract is below 20 grammes.

To secure constant results by distillation the author recommends that sufficient distilled water should be placed in the receiving flask to cover the mouth of the condenser tube (which is lengthened for this purpose) at the commencement of distillation, the receiver being afterwards inclined at an angle, so as to free the mouth of the tube and avoid complete absorption.

This arrangement obviates the necessity for wet cloths or filter papers to condense the alcoholic vapour.

C. S.

ORGANIC ANALYSIS.

Determination of the Heat of Bromination in Oils. H. W. Wiley. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 378-385.)—After speaking in general terms on the value of Hehner and Mitchell's process (*ANALYST*, xx., 146), Dr. Wiley describes some modifications which he has adopted with the view of rendering it more easy of application. The bromine is dissolved in the chloroform in the proportion of 1 volume

in 4, and this solution used instead of the liquid bromine. The bromine solution is placed in an Erlenmeyer flask with a side tubulure near the top, on which is fixed a rubber bulb. Through the stopper of the flask a pipette is passed and made air-tight, and the bromine solution can then be readily blown up into the pipette by compressing the bulb.

Another modification consists in dissolving a larger amount of the fat in chloroform and taking aliquot portions of the solution, thus allowing several determinations to be made on the same sample.

The calorimeter employed is a tube about 40 cm. in length and $1\frac{1}{2}$ cm. in internal diameter, fitted air-tight by means of a rubber cork into a drying jar with a side tubulure. To secure insulation of the inner tube, air is withdrawn from the drying flask through the side tubulure.

The author has found carbon tetrachloride preferable to chloroform as a solvent, by reason of its greater stability and higher boiling-point. The rise of temperature, however, is slightly less than in the case of chloroform solutions.

As to the ratio between the rise of temperature and the Hübl number, stress is laid on the point that the factor 5.5 is not universally applicable, but that the factor must be determined separately for every system of apparatus and solvent employed.*

W. J. S.

Determination of the Acidity of Pyroligneous Products. Scheurer-Kestner. (*Comptes rendus*, cxxii., p. 619).—Among the bodies associated with acetic acid in crude pyroligneous acid are phenols and acetic ethers, notably methyl acetate (containing 15 to 17 per cent. of the total acid), and it therefore becomes necessary both to remove the phenols, which vitiate the results of titration, and to determine the amount of acid in the methyl acetate, since this latter escapes observation when the ordinary method is pursued. Both objects may be effected by distillation over phosphoric acid, which retains the phenols and decomposes the methyl acetate. Twenty grammes of the crude acid are distilled in a flask along with 50 grammes of phosphoric acid (15° B., S. G. 1.116), until merely a small quantity of liquid remains. An addition of 20 c.c. of water is then made and the distillation continued, this operation being repeated once more. When the distillate which comes over is nearly neutral to test-paper, it is ready for titration by sodium hydroxide, phenolphthalein being the most suitable indicator.

This method gives results 10 per cent. below those obtained by the ordinary process, the error in the latter being due to the reaction of the phenols. It is also suitable for application to acetate of iron or alumina, being, in fact, a development of the Fresenius method for the analysis of calcium acetate.

C. S.

Technical Analysis of Asphaltum. S. P. Sadtler. (*Jour. Franklin Inst.*, 1895 [5], p. 383).—Criticising the method described by Linton (*ANALYST*, xx., p. 41), Sadtler proposes to discard the use of turpentine, and replace the petroleum spirit by acetone for extracting the petroleum, retaining chloroform as a solvent for the asphaltene.

* This was specially stated in Messrs. Hehner and Mitchell's paper.

One to two grammes of the asphalt are carefully mixed with some 10 grammes of fine white sand in a Gooch crucible containing an asbestos filter, the apparatus having been previously dried at 100° C. till constant. The drying is repeated with the asphalt, and the decrease in weight is designated "moisture and loss at 100°." The crucible and contents are inserted in a small percolator contained in a larger one connected with a flask holding the acetone, and with a vertical condenser. Heat is applied (a sand-bath being preferred) and extraction continued until the loss has decreased to 0.5—1 milligramme per hour, a result generally attained at the end of some twelve hours. The loss by this extraction is considered as "petrolene." Chloroform is then substituted for the acetone, and the extraction of the "asphaltene" effected in about eight hours. The residual matter in the crucible represents organic non-bitumen and mineral substances; the latter may be estimated by incineration and weighing.

C. S.

Volumetric determination of Uric Acid. G. Denigés. (*Bull. Soc. Pharm. Bordeaux*, 1896, 75; through *Ann. de Chim. Analyt.*, I. [8], 148.)—The method proposed is by throwing down the uric acid by an excess of cuprous hyposulphite, and titrating the copper by the cyanide method.

The following reagents are required :

1. 160 grammes anhydrous sodium carbonate,
2. 100 " crystallised sodium hyposulphite,
- 100 " sodium potassium tartrate,
3. 40 " pure crystallized copper sulphate,
- 10 drops sulphuric acid of 66° B. (S. G. 1.842),

each made up to 1,000 grammes of solution by distilled water.

The phosphates of the alkaline earths are thrown down from 100 c.c. of urine by 10 c.c. of solution No. 1, and the filtrate mixed with 40 c.c. of No. 2 and 10 c.c. of No. 3. The supernatant liquid is decanted at the end of 10 minutes and aspirated through a flat filter, making sure that sufficient of the reagent is added to bring down the whole of the precipitable matter. The filter must be well washed repeatedly with a thin stream of water to collect the whole of the precipitate at the centre, and left to drain after each operation. When diabetic urine is under examination, washing must be continued until all the sugar is removed.

The precipitate is then washed with boiling water into a porcelain capsule, and hydrochloric acid (0.5 to 1.5 c.c.) added, followed by sodium hypobromite, or bromine water, drop by drop, until the copper urate is dissolved, and the liquid becomes permanently yellow, or yellow-green.

The solution which should not exceed 40 c.c., is heated to boiling, 10 c.c. of ammonia added, and whilst the boiling is brisk and uninterrupted, decinormal potassium cyanide solution added, drop by drop. Towards the end point the boiling must be kept brisk and the reagent added only every third or fourth second, until the blue coloration of the solution disappears.

The amount of copper is calculated by the formula :

$$(n - 0.1) \times 0.594 \times 0.00635$$

n being the number of c.c. of cyanide solution.

$$(n - 0.1)0.1 + (n - 0.1)0.01.$$

Estimation of Fusel-oil in Rectified Spirit. A. Stutzer and R. Maul.

The following examples are given :

	Height.	Difference.
	c.c.	c.c.
With pure alcohol	20.59	—
Addition of 0.01 per cent. amyl alcohol	20.63	0.04
" 0.10 " " 	21.08	0.44
" 0.20 " " 	21.48	0.89

Hence it appears that a difference in volume of 0.1 c.c. corresponds to 0.022472 per cent. amyl alcohol in 30 per cent. spirit, or 0.075 per cent. in 100 per cent. spirit,

and thus, by concentrating the amyl alcohol, as described above, 0.005 per cent. by volume in 100 per cent. spirit can be accurately determined. C. A. M.

Determination of Albumin in Urine. G. Mercier. (*Ann. de Chimie Analyt.* i., 125.)—The most satisfactory results are obtained when the quantity of albumin does not exceed 100 to 150 milligrammes. It therefore becomes necessary, when the percentage of albumin is high, to take only a small quantity (10 c.c. or less) of urine, and to make up to 50 c.c. with distilled water. A frequent result of this dilution is imperfect coagulation, and the escape of portions of the albumin along with the filtrate. This defect may be remedied by restoring to the solution the proper balance of saline matter by adding about 1 gramme of sodium chloride. In such cases the coagulum must be thoroughly washed with boiling distilled water until no trace of chloride can be detected in the washings. C. S.

Estimation of Para-Sulphanilic Acid. Karl Brenzinger. (*Zeit. ang. Chem.*, 1896, 131-133.)—This method depends on the decomposition of the sulpho group by means of bromine, and the subsequent estimation of the liberated sulphuric acid. It is necessary to avoid a large excess of bromine, as otherwise the barium sulphate precipitate will be contaminated by sulpho acids.

In the experiments with pure water-free para-sulphanilic acid, one-tenth of a molecule (17.3 grammes) was dissolved in a litre of water, and the solution, if alkaline, made slightly acid with hydrochloric acid. Portions of 100 c.c. were then treated with saturated bromine water, until after standing fifteen or twenty minutes there was a slight excess of bromine when they were tested with potassium iodide starch-paper. After twenty minutes pure soda solution was added to slightly alkaline reaction to remove the slight excess of bromine, the liquids filtered, and the sulphuric acid in the filtrates determined in the usual manner. Warming the liquid before filtration was advantageous, but not essential, in the case of pure para-sulphanilic acid, but where metanilic acid was present the precipitation, filtration, and washing had all to be done hot. In impure samples the sulphuric acid was determined first, and the amount deducted from that subsequently obtained after bromination.

EXAMPLES.

Pure Acid taken.	BaSO ₄ found.	BaSO ₄ calculated.	Para-sulphanilic Acid.	Per cent.
1.73	2.3294	2.330	1.7291	99.95
1.73	2.3250	2.330	1.7253	99.71

IMPURE SAMPLE.

1.73 grammes yielded 0.673 gramme BaSO₄ before bromination.

“ “ 2.4402 grammes BaSO₄ after bromination.

Difference = 1.6528 gramme BaSO₄ = 79.3 per cent. para-sulphanilic acid.

Experiments made with mixtures of pure metanilic acid and pure para-sulphanilic acid gave the following results :

Para-sulphanilic Acid added. Per cent.	BaSO ₄ obtained. Grammes.	=	Para-sulphanilic Acid. Per cent.
0.2	0.0083		0.35
1.0	0.0305		1.3
5.0	0.1160		4.98

In three specimens of impure metanilic acid the amount of para-sulphanilic acid found was 4.93, 4.47, and 4.27 per cent. respectively. In the absence of other compounds, on which nitrites could act, the sum of both acids could be obtained by diazotizing, and the amount of metanilic acid by deducting from this the quantity of para-sulphanilic acid found by the method described above.

C. A. M.

Estimation of Aniline and Toluidine in presence of small quantities of each other. P. Dobriner and W. Sobranz. (*Zeit. anal. Chem.*, 1895, xxxiv., 734-740.)—The method described by the authors is an adaptation of Reinhardt's process (*Chem. Zeit.*, xvii., 413), and is based on the fact that aniline forms a tribromo-derivative, while *o*- and *p*-toluidine give dibromo-derivatives. Reinhardt's directions are slightly modified, hydrobromic acid being replaced by potassium bromide and hydrochloric acid, and starch iodide paper used as the indicator. The brominating solution is standardized with pure aniline, and the value thus obtained multiplied by $\frac{160.5}{93}$ gives the toluidine value. When water is absent, one titration will give the amount of the aniline and toluidines in a commercial oil. Where a = weight of oil, x = amount of aniline, t = value of brominating solution standardized on pure aniline, and v = No. of c.c. of solution used :—

$$x = 2.3777vt - 1.3777a,$$

and $a - x$ gives the amount of toluidine in the oil. When only a small quantity of aniline is present, as in commercial toluidine, the results are too high. In that case, the brominating solution should be standardized with pure toluidine, and the aniline value t obtained by multiplying by $\frac{93}{160.5}$. Or (as is preferable) the solution may be standardized with both pure aniline and pure toluidine, and the amount of aniline calculated by the equation

$$\frac{x}{t} + \frac{x - a}{T} = v$$

where t and T represent the standardization values. In making a determination, about 150 c.c. of the brominating solution (1 litre = 8 grammes aniline) are used, and the readings taken at 15° C. The test analyses of mixtures quoted are satisfactory.

C. A. M.

Estimation of Moisture in Aniline and in Ortho- and Para-Toluidines. P. Dobriner and W. Sobranz. (*Zeit. anal. Chem.*, 1895, xxxiv., 740-742.)—Reinhardt's method (see preceding abstract) is applied to this estimation by brominating

equal weights of the moist and dried substances, and calculating the percentage of moisture F by the formula

$$a : b = (100 - F) : 100,$$

where a = number of c.c. absorbed by the wet substance

" b = " " " " " dry "

Treatment with fused potassium carbonate renders aniline and *o*-toluidine anhydrous, while *p*-toluidine may be distilled, and the residue, after distilling 10 per cent., taken as dry.

C. A. M.

INORGANIC ANALYSIS.

Estimation of Potash by the Carnot Method. Ed. Goutal. (*Ann. de Chimie Analyt.*, i., 89-91.)—The following particulars are given of the method as performed at the laboratory of the Ecole Nationale des Mines, under the direction of M. Carnot himself.

The reagents are prepared thus :

1. *Bismuth Chloride Solution*.—100 grammes of bismuth subnitrate dissolved by exactly the necessary amount of hydrochloric acid, and made up to 1 litre with 95 per cent. alcohol. If any turbidity ensues—due to the precipitation of a sub-salt—it should be redissolved by a few drops of hydrochloric acid.

2. *Calcium Thiosulphate Solution*.—This should contain 200 grammes of the pure crystallized salt per litre. The thiosulphate must be freshly prepared, exhibit no tinge of yellow, and be properly crystallized.

3. *Iodine Solution*.—A standard solution containing 26.96 grammes of pure iodine dissolved by the aid of about 40 grammes of pure potassium iodide.

4. *Sodium Thiosulphate Solution*.—Standardized to correspond to the iodine solution—i.e., containing 52.64 grammes of the crystallized salt per litre.

The substance is dissolved in 8 c.c. of water, and the solution (which may include a trace of free hydrochloric acid) should not contain more than 700 milligrammes of potash. To obtain an average sample, in the case of more or less homogeneous complex bodies, it is better to dissolve from 20 to 30 grammes in the proportion of 1 gramme of substance to 8 c.c. of water.

A mixture of the reagents is then prepared by adding together in the following order : 20 c.c. of the alcoholic bismuth solution, 20 c.c. of the calcium thiosulphate solution, and 200 c.c. of 95 per cent. alcohol ; this should produce a perfectly clear liquid. When the substance contains less than 300 milligrammes of potash, one half the foregoing quantities of bismuth and thiosulphate solutions, and 150 c.c. of alcohol will be sufficient.

The reagent is poured into the potash solution, and, after vigorous agitation left at rest for half an hour, by which time the precipitate of double thiosulphate of bismuth and potassium will have fallen. On decanting the liquid, the precipitate is thrown on to a filter, and carefully washed with 95 per cent. alcohol. Quick filtration is desirable, but the aspirator need not be resorted to if the paper is good and filters freely.

The double salt is dissolved by washing with cold water on the filter, and, after

the addition of a little fresh starch paste and 2 or 3 c.c. of hydrochloric acid, iodine solution is added from a burette until a dark brownish-green coloration (passing suddenly from pale yellow) is produced. Should the end-point have been overstepped, the sodium thiosulphate solution is used. Each c.c. of iodine solution corresponds to 1 centigramme of potash.

The method is very exact and rapid, but requires the observance of the following precautions :

The calcium thiosulphate *must* be freshly prepared ; the bismuth solution must contain exactly 100 grammes of subnitrate per litre ; 95 per cent. alcohol alone should be used, as the precipitate is partly soluble in weaker spirit ; and the mixed solution must be *slightly* acid. It is also important that the thiosulphate should not be in excess as compared with the bismuth, or a portion of it will be thrown down by the alcohol and vitiate the result. If, however, all the conditions be observed, a careful operator will be able to determine with ease potash existing indifferently as chloride, nitrate, carbonate, phosphate, or even sulphate, without the necessity for the previous removal of such associated bases as soda, lithia, ammonia, lime, magnesia, iron or manganese oxides, etc.

C. S.

Lösche's Process for the Estimation of Potassium. H. Haefcke. (*Chem. Zeit.*, 1896, xx., 88.)—This process (*ANALYST*, xxi., 81), really devised by Mehns, is by no means original. A precisely identical method was described by De Roode in 1895 ; and one essential feature of it—the removal of *sodium* sulphate from a precipitate of potassium platinochloride by the action of ammonium chloride solution—is due to Finkener, who published it in 1867. The latter noted that the ammonium chloride decomposed up to 5 per cent. of the potassium salt, especially in presence of hydrochloric acid, even at ordinary temperatures, and during one hour's action ; so that in Lindo and Gladding's method—a standard commercial process in America—the solution is treated for twenty-four hours before use with the potassium platinochloride, in order to saturate it, although Breyer and Schweitzer have pointed out that this device is not sufficient to prevent further action. The claim that *potassium* sulphate is converted into the double platinum salt by washing with ammonium chloride under conditions that leave the corresponding sodium compound unattacked has not been substantiated, and, indeed, seems incredible. In short, the process cannot be considered reliable.

F. H. L.

The Estimation of Potassium. (1) A. Prager, (2) R. Ruer, (3) E. Bauer. (*Chem. Zeit.*, 1896, xx., 269 and 270.)—In making use of the process that Fresenius has described as most suitable when the potassium in the sample exists only as sulphate, Prager considers that the purity of the precipitated platinum salt can only be relied on when it is obtained in the crystalline condition. Even extracted filter-papers contain matter soluble in 80 per cent. alcohol and hot water ; and it is absolutely necessary, therefore, to treat them with these liquids before determining their original weight. (Fresenius prefers an asbestos filter.) The author's process is as follows : The sulphuric acid is removed with the smallest possible excess of barium

chloride, and after filtration the liquid is made up to 75 c.c., the platinum solution added, and the whole evaporated on a water-bath, which is not allowed to boil. Directly crystals appear on the surface, the liquid is cooled, and, after removal of the deposit, evaporated slowly down to 5 c.c. It is again cooled, diluted with 20 c.c. of 96 per cent. alcohol, stirred well, and allowed to stand for a time. The whole of the precipitate is then brought on the prepared filter by means of 80 per cent. spirit, and it is finally dried in a well-ventilated oven at 110° C. The double chloride should afterwards be treated with a small quantity of cold water to test its purity. The factor 0.19308 is to be used to convert the platinochloride into potassium oxide.

Ruer finds that the ordinary practice in many potassium works of heating the double salt for only twenty minutes to 130° C. gives a product which may for most practical purposes be considered dry and fit for weighing. It nevertheless contains sufficient moisture to render the usual factor of 0.3056 (to calculate it into potassium chloride) too high. Fresenius has endorsed this figure; but his experiments were carried out on precipitates dried for at least twelve hours, and he insists on the necessity of heating till constant weight. To avoid this loss of time, the author suggests drying the platinum salt at 130° for half an hour, and making use of the factor 0.304, otherwise the results, although perfectly concordant, are uniformly 0.5 per cent. above the correct amount.

Bauer suggests that, instead of drying and weighing the precipitate on the filter, it should be dissolved in hot water, and the solution allowed to run through the paper into a tared platinum basin. The filtrate is then evaporated and dried at 120° C. This improvement obviates the danger of the platinochloride being partially reduced by careless drying, does away with the unsatisfactory process of using a weighed filter-paper, and removes any insoluble matter in the precipitate, which may be due either to traces of barium or to the presence of platinous chloride in the reagent. In this manner also the platinum residues are kept pure and free from organic matter.

F. H. L.

Examination of the Methods for the Determination of Manganese in Iron and Steel. L. Rürup. (*Chem. Zeit.*, 1896, xx., 285 and 337.)—The author has re-investigated six different processes for this purpose, testing them on crucible, Bessemer, and Martin steel, and on Thomas cast-iron. Care was taken that the borings should be uniform in composition by drilling the samples only at one spot, and by preparing at one operation sufficient material for all the tests. The gravimetric processes employed were: (1) Estimation as manganese sulphide; (2) precipitation by bromine, and weighing as Mn_2O_4 ; (3) Ford's process. The volumetric methods were: (4) Volhard's; (5) precipitation of the iron with sodium sulphate followed by titration of the manganese; (6) Hampe's chlorate process.

All the samples were examined six times by each method, and the results were almost identical in every case, showing a perfectly satisfactory agreement with the theoretical amount of manganese present. As regards the time occupied in carrying them out, (5) and (6) are the quickest, consuming only $1\frac{1}{2}$ to $1\frac{1}{4}$ hours, the others requiring longer, up to three hours in the case of (1).

(5) is in use in Krupp's laboratory, and is carried out as follows: 4 grammes of the borings are dissolved in 75 c.c. of 1.2 nitric and 10 c.c. of hydrochloric acid; the solution is poured into a litre flask, sodium carbonate added till the liquid is just rendered faintly opaque, then 1.8 grammes of sodium sulphate is introduced, the whole made up to the mark, and allowed to stand without heating till the precipitate has subsided. It is filtered into a 750 c.c. flask, boiled with 15 grammes of zinc sulphate, and titrated with permanganate.

F. H. L.

A Gravimetric Method of Estimating Phosphoric Acid as Ammonium Phospho-molybdate. T. S. Gladding. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 28-27.)—To the solution of phosphoric acid 25 c.c. of strong ammonia (0.900 specific gravity) are added, and then nitric acid (1.42 specific gravity) to acid reaction. The beaker is placed in a water-bath maintained at 50° C., and the ordinary 10 per cent. acid molybdate solution added at the rate of about three drops per second, with constant stirring, until about 10 c.c. in excess. After remaining ten minutes in the bath, the liquid is filtered through a weighed filter-paper, previously dried at 105° C., and the filtrate tested with more molybdate solution. The precipitate is washed three times by decantation, and three times on the paper with dilute nitric acid (1:100), and once with distilled water. The paper and contents are drained on filter-paper, and then dried at 105° C., until the weight becomes constant. For drying at this temperature, the author found a water-bath surrounded with dilute glycerine (1.160 specific gravity), boiling at 110° C., the most reliable means. As thus obtained, the yellow precipitate is of very uniform composition, and free from separated molybdic oxide or iron. Multiplication by the factor 0.0376 gives the amount of phosphoric acid it contains.

The results obtained with a solution of pure microcosmic salt, containing 0.5 grammes in 50 c.c., were 34.06 to 34.10 per cent. of phosphorus pentoxide by this method, and 34.05 to 34.09 per cent. by precipitation with magnesia mixture. Four determinations were made by each method. To test the applicability of the method to the determination of very small amounts, the above solution was diluted to one-tenth:

	Taken.	Yellow Salt Obtained.	P ₂ O ₅ Obtained.	P ₂ O ₅ Theoretical.
1.	10 c.c.	...	0.00342	...
2.	1 "	...	0.00037	...
			0.00034	

Comparative tests with a number of fertilizers also gave closely agreeing results, the phosphoric acid being as follows:

Official Method.			New Method.
Per cent.			Per cent.
28.80	28.87
2.68	2.70
12.03	12.00
28.30	28.33
15.64	15.70
15.04	15.00
15.19	15.23
29.16	29.23

In each case 0.250 gramme was used for precipitation, and an excess of about 10 c.c. (not more) of the molybdate solution added.

C. A. M.

A Modified Ammonium Molybdate Solution. A. L. Winton. (*Jour. Amer. Chem. Soc.*, 1896, xviii., pp. 445-446.)—The following formula is recommended for the preparation of a solution containing the same proportion of molybdic acid and free nitric acid as the solution of Fresenius, but having 15 grammes more ammonium nitrate in 50 c.c. :

1. Dissolve 1,000 grammes of molybdic acid in 4,160 c.c. of a mixture of one part of concentrated ammonia (sp. gr. 0.90) and two of water.

2. Dissolve 5,300 grammes of ammonium nitrate in a mixture of 6,250 c.c. of concentrated nitric acid (sp. gr. 1.4) and 3,090 c.c. of water.

Add I. to II. slowly with constant stirring, and decant the clear liquid after standing for a few days in a warm place.

The use of this solution obviates the necessity for the separate addition of ammonium nitrate, which is often employed to facilitate the separation of the molybdic precipitate and to shorten the time of digestion.

C. A. M.

Analysis of a Mixture of Chlorides, Chlorates, and Perchlorates. Ad. Carnot. (*Comptes rendus*, cxxii., p. 452.)—Perchlorates are found along with chlorides and chlorates in the products of the calcination of chlorates. Hypochlorites are only produced in the cold or by wet methods; but in such cases no perchlorates are formed, nor can the latter be reduced by the usual reagents in solution, dry heat being necessary to accomplish this result.

In analysing such mixtures, the chlorides and chlorates are estimated first, by titrating one portion of the solution for the chlorides with argentic nitrate and ammonium thiocyanate, and the other part for the total chlorine after reduction of the chlorates by the aid of ferrous sulphate; or, as an alternative method, both titrations can be performed on the same liquid, the chlorides first—with sodium arseniate as indicator in preference to potassium chromate, which would interfere with the subsequent reaction—and then the total chlorine after reduction of the chlorates.

Finally, the perchlorates are determined by heating the powdered substance, mixed with four or five times its weight of purified quartz-sand, in a platinum crucible, the mixture being covered with a layer of the same sand 1 or 2 c.m. deep. The bottom of the crucible is kept at a red heat for about twenty to thirty minutes, and this is sufficient to completely reduce the chlorates and perchlorates, volatilization of the chloride being prevented by the condensing effect of the upper layer of sand. An aqueous solution is then made, the total chlorides titrated as before, and the perchlorate estimated by difference.

C. S.

Volumetric Determination of Mixtures of Chlorides, Hypochlorites and Chlorates. Ad. Carnot. (*Comptes rendus*, cxxii., 449.)—These mixtures are met with in the products resulting from the action of chlorine on the hydroxides of the alkalis or alkaline earths, chlorate being always present in bleaching powder in small quantities, increasing with the age of the product, especially when the lime used contained carbonate. The same salts are also formed when chloride of sodium is subjected to electrolysis.

A single solution suffices for the estimation of all three substances. The hypochlorite is determined by titration with sodium arsenite, which does not reduce the chlorate in neutral or alkaline solution, the end point being gauged by testing a drop of the solution with potassium iodide and starch. The liquid, which now only contains the chlorates and chlorides, is acidified with a little sulphuric acid and heated to nearly 100° C., with about twenty times as much ferrous ammonium sulphate as the amount of chlorate suspected. Then a solution of 5 c.c. of sulphuric acid in 15 c.c. of water is added drop by drop, and, after being left to cool in the flask (closed to prevent access of air), the excess of ferrous salt is titrated by permanganate. This allows the amount of chlorate to be calculated, and, after decolorizing the solution by a little ferrous sulphate, the total chlorine is determined by adding a measured quantity of argentic nitrate, and titrating back the excess by means of ammonium thiocyanate. The end point is indicated by the formation of red ferric thiocyanate, which occurs as soon as all the silver salt has entered into combination. C. S.

The Iodometric Estimation of Selenious and Selenic Acids. F. A. Gooch and A. W. Peirce. (*Zeit. f. An. Chem.*, xi., 249.) This is an improvement on methods previously published by one of the authors (Gooch and Reynolds, *Zeit. f. An. Chem.*, x., 248) in which selenious acid is heated with acidulated potassium iodide, and the iodine estimated volumetrically in the distillate and residue.

To avoid the double estimation of iodine the authors now propose a method based on a reaction previously employed for the estimation of chlorates (Gooch and Smith, *Amer. Journ. Sci. [Sill.]*, xlii., 220). If an acid solution of potassium iodide be heated with excess of arsenic acid, all the iodine is liberated, a corresponding proportion of arsenic acid being reduced to arsenious acid. By prolonged heating the iodine is completely volatilized, and the residue, after being made alkaline, is titrated with standard iodine, of which the quantity required is a measure of the arsenious acid formed.

If in addition to arsenic acid a more easily reducible substance is present, such as a chlorate, or selenious acid, it gets reduced in preference to the arsenic acid, and consequently less arsenious acid is formed and less iodine is required in the final titration. The difference between the iodine solution used up under these circumstances, and what would have been required had arsenic acid alone been initially present, indicates the quantity of the chlorate or selenious acid.

The practical details are as follows:—A weighed portion of the selenious acid is introduced into a 300 c.c. Erlenmeyer flask along with a known weight of potassium iodide (which should be in excess of what is theoretically required) in a solution of

which the strength in terms of arsenious acid has been accurately ascertained. Finally, a solution containing about 2 grammes of acid potassium arsenate and 20 c.c. of 50 per cent. sulphuric acid is added.

To prevent loss by spurting a short calcium chloride tube is allowed to rest with its wide end in the neck of the flask, and to avoid bumping some pieces of pipe-clay are put into the liquid, which is then boiled until the original volume of about 100 c.c. is reduced to 35.

The residue is cooled and neutralized with caustic potash. 20 c.c. concentrated solution of sodium bicarbonate, and some starch solution are then added and the titration with normal iodine performed. The difference between the amount thus required, and the larger volume which would have been needed in the absence of selenious acid (due to the arsenic acid alone being reduced), indicates the proportion of selenious acid.

To estimate selenic acid it must first be reduced to selenious acid, which is best done by means of potassium bromide and sulphuric acid as previously shown (*Zeit. f. An. Chem.*, x., 253) the use of hydrochloric acid being inadmissible on account of the arsenic trichloride which would be afterwards formed.

NOTE BY ABTRACTOR.—The authors appear only to have tried the process on the pure substances.

C. H. C.

Acidimetric Determination of Zinc. L. L. de Koninck. (*Mon. Scient.*, 1896, 180).—Working with a one-fifth normal solution of zinc potassium sulphate, and pure titrated potash, to test the rival opinions of Barthe and Lescœur on the composition of the precipitate, the author finds that this varies with the temperature, being $7\text{ZnO}, 2\text{ZnSO}_4$ when the operation is performed in the cold, and $17\text{ZnO}, 3\text{ZnSO}_4$ at 65°C . At 100°C . the decomposition of the zinc salt is complete within 1 or 2 per cent.; the final reaction is slow, the phenolphthalein losing its colour gradually. An excess of alkali is necessary to produce complete decomposition, and the only satisfactory way of titrating this back is by operating at boiling temperature and checking the colour by the aid of a duplicate solution containing no phenolphthalein.

The method, pursued in this manner, is exact, but is not suitable for ores. C. S.

The Quantitative Determination of Hydrogen. E. D. Campbell and E. B. Hart. (*Amer. Chem. Jour.*, 1896, xviii., 294-298).—In 1894 F. C. Phillips (*Amer. Chem. Jour.*, xvi., 256) proposed to determine hydrogen by absorption with dry palladious chloride, but was unable to obtain satisfactory results. The authors have devised the following method, based on the same principle, but using a solution of palladious chloride. This is prepared by dissolving 5 grammes of palladium-wire in 30 c.c. of hydrochloric acid, to which has been added 1.2 c.c. of nitric acid. The solution is evaporated just to dryness on the water-bath, and the residue dissolved by warming with 5 c.c. of hydrochloric acid (specific gravity 1.20), and 20 to 25 c.c. of water. This solution, when diluted to 750 c.c. gives a nearly neutral solution containing about 1 per cent. of palladious chloride.

The pipette employed is the ordinary Hempel absorption-pipette, which is made so as to be readily detachable from its stand. In making an estimation, the solution is first introduced into the pipette, then the gas to be analysed, from which everything except hydrogen, paraffins, and nitrogen has been removed, and finally sufficient water to completely fill the capillary.

The pipette is then disconnected, the top being first closed with a pinch-cock, and placed in a hot water-bath for two hours, which is sufficient time for complete absorption, unless the amount of hydrogen exceeds 65 c.c., or the pipette has been previously used. After the pipette has been used for the absorption of about 100 c.c., the remaining solution should be evaporated just to dryness on the water-bath, 5 to 6 c.c. of hydrochloric acid, 4 to 5 c.c. of nitric acid, and a little water added, and the evaporation repeated. The dry palladious chloride is dissolved by adding 2 c.c. of hydrochloric acid, and warming with a small amount of water, and the whole diluted to the original volume and returned to the stock-bottle.

The experiments summarized below show the accuracy of the method, all volumes of gas being reduced to standard temperature and pressure :

Volumes taken.		Strength of Palladious Chloride Solution.	Time of Heating.	Residue, c.c.
Hydrogen, c.c.	Nitrogen, c.c.			
18.7	80.3	1 per cent.	To 50° C., overnight.	80.3
53.7	23.8	" "	Water-bath, 90 minutes.	23.8
1.07	52.23	" "	" "	52.62
1.27	70.8	2 per cent.	Water-bath, overnight.	70.84

From these and other experiments the author concludes that a 2 per cent. solution of palladious chloride has very little advantage over a 1 per cent. solution. With strongly acid solutions absorption was retarded.

C. A. M.

The Estimation of Pyrrhotite in Pyrites Ores. E. F. Cone. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 404-406.)—In the manufacture of sulphuric acid from pyrites ores, it is found impossible to burn off the greater part of sulphur present in the form of pyrrhotite (Fe_7S_8), which is often a constituent of American ores. The estimation of pyrrhotite sulphur is therefore of considerable importance, and for this purpose the author describes the following process, which is based on the fact that Fe_7S_8 is magnetic, while pyrites is non-magnetic. The ground ore is passed through a sixty-mesh sieve, and 13.74 grammes weighed out and spread on glazed paper. A magnet is then passed through the powder, and the magnetic portions separated. This is repeated several times, the separated portion ground in an agate mortar, and the sulphur estimated gravimetrically by oxidation with nitric and bromo-hydrochloric acids. The weight of barium sulphate obtained in grammes is the percentage of sulphur present as pyrrhotite. The method is accurate to within one-fifth per cent., but the ore must not be finer than will pass through a sixty-mesh sieve, or the results will be unreliable. Examples :

A SAMPLE OF ORE CONTAINING PYRRHOTITE.

	Per cent.			
Total sulphur ...	35.07	Sulphur present as Fe_7S_8		24.14
„ iron ...	57.50	Iron „ Fe_7S_8		36.96
Oxygen as Fe_3O_4 ...	4.26	„ „ FeS_2		9.34
Copper ...	0.25	Sulphur „ FeS_2		10.68
Insoluble matter ...	2.78	Iron „ Fe_3O_4		11.20
	99.86			

A small proportion of this ore was added to pyrites containing no pyrrhotite, so that the mixture contained 1.20 per cent. of sulphur as Fe_7S_8 . This mixture was analysed for pyrrhotite sulphur, using different weights. Some of the results obtained were :

Grammes.		BaSO_4		Per cent. Sulphur as Fe_7S_8 .
5.00	gave	0.4120	=	1.13
13.74	„	1.13	=	1.13
25.00	„	2.1570	=	1.18

C. A. M.

CORRESPONDENCE.

THE EXAMINATION OF WATER.

To the Editor of THE ANALYST.

SIR,—In order, if possible, to bring bacteriologists and chemists into line as to the relative value of their respective methods of water-examination, it is necessary to keep this subject constantly before the profession. I, therefore, offer no apology for drawing attention to some recent remarks made by Dr. Klein in the course of his Harben Lectures, as reported in the *Times* of June 9, 1896. They are as follows : “The chances of finding the typhoid bacillus in ordinary cases, where it was only present in small quantities, were *exceedingly remote*. It was like looking for a needle in a rick of hay. Statements, therefore, such as were repeatedly made, to the effect that certain samples of water, milk, or other materials did not contain the typhoid bacillus were *quite valueless*. The *only safe rule* was to regard any fluids in which the presence of sewage organisms has been demonstrated with suspicion, whether the typhoid bacillus was found or not.” The italics are my own. Here, then, we have an emphatic declaration by one of our leading bacteriologists that the outcry against the chemical analysis of water, because it could not detect the presence of pathogenic organisms, was a vain and foolish one. The only question now requiring consideration in connection with this matter as I pointed out in my note read before our Society in March, 1895, is the following : “How can sewage pollution be most certainly detected, chemically or biologically ?” I have given my answer in the note mentioned above, and should now like, with your permission, to invite discussion in your pages on this most important point, as it seems to me high time that this vexed question should be settled one way or another in the interest of the public as well as of the profession.

Yours, etc.,

A. DUFRÉ.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

COMMERCIAL PRUSSIAN BLUE.

By ERNEST J. PARRY, B.Sc., F.I.C., AND JOHN HENRY COSTE, F.I.C.

(Read at the Meeting, June 3, 1896.)

IN G. Hurst's well-known book, "Colours, Oils, and Varnishes," a scheme for the analysis of colours containing Prussian blue is given as follows: "A known weight of the sample should be boiled in caustic soda until the blue is completely decomposed. The mixture is then filtered and the brown residue well washed with hot water until it is quite free from alkali. Next, it is treated with hydrochloric acid until the brown oxide of iron has been dissolved. Lastly, it is filtered, and to the filtrate is added ammonia in slight excess. The precipitate of oxide of iron thus formed is filtered off, washed with water, dried and burnt in a crucible, and then weighed. *The weight of the oxide of iron multiplied by 2.212 gives the amount of Prussian blue in the sample taken.*"

As a matter of fact, under these conditions Prussian blue yields about 28 per cent. of Fe_2O_3 , so that estimations based on Hurst's statement would fall short of the truth by at least 35 per cent. Hurst is here evidently under the impression that all the iron in the pigment is precipitated by treatment with alkali, whereas, of course, all in the form of iron-cyanogen radicle is lost in the filtrate as alkaline ferrocyanide.

The following is given as the complete analysis* of a sample of Chinese blue, one of the best varieties of Prussian blue:

Water	4.487
Oxide of iron	52.055
Cyanogen	43.508

100.000

The method here employed is evident. Briefly, we presume it involves the determination of the moisture and the ash. Since the pigment contains no oxygen, even if the ash were entirely oxide of iron, the iron present is only 70 per cent. of the amount of oxide, and the cyanogen, evidently taken by difference, would be correspondingly higher.

We have found, and Dr. Williamson† in every case found, that alkali was present in so-called Prussian blue, not merely as alkaline salts not washed away completely, but as an integral part of the cyanide. This alkali does not figure in the above analysis.

* These figures add up to 100.05, evidently by a clerical error.

† Chem. Soc. Mem., 1845-46, 125.

As we are frequently called upon to examine commercial samples of Prussian blue, and of pigments containing this substance, we have considered it desirable to make a more complete examination than the method given above would afford of specimens of blue of known origin, with a view to getting some more definite data for basing conclusions as to the purity of samples of unknown make.

It appeared to us that, more especially in the case of complex pigments ground in oil, the determination of the nitrogen would give valuable information as to the amount of Prussian blue present.

Dyer has shown (*Chem. Soc. Jour.*, 1896, 811) that ferrocyanides and ferricyanides yield the whole of their nitrogen as ammonia when treated by the Kjeldahl-Gunning-Arnold method. We found that potassium ferrocyanide boiled with sulphuric acid for a few minutes gave up all its nitrogen as ammonia. A determination of the water of hydration (lost at 180° to 200° C.) was also made. The results were :

			Found.	Theory.
N	19.64	19.95
H ₂ O	12.79	12.71

We therefore felt justified in assuming that equally correct results would be obtained in the case of the allied compounds contained in commercial Prussian blue, an expectation which was fully justified by the agreement of the nitrogen determinations with the other figures obtained.

The following is briefly the scheme of analysis adopted by us :

The moisture is determined at 100°. The water of combination—if the term is here justifiable—can scarcely be determined unless the blue is “burned” and the water collected, as we made several attempts to drive off the water in a current of hot air, gradually raising the temperature until at 230° C., when water appeared to be still coming off, the pigment was decomposed with the formation of ferric oxide. Dr. Williamson, in his classical paper, “On the Blue Compounds of Cyanogen and Iron,”* determined the total water in these pigments by combustion with plumbic chromate. This is apparently the only way of obtaining a correct result, as, on heating, part of the hydrogen is evolved as hydrocyanic acid, the oxygen of the water going to form ferric oxide, as stated. The nitrogen was determined as above described, and was found to differ within small limits. A weighed quantity was ignited, care being taken that the temperature was sufficiently high to decompose the last traces of the blue, but not too high to render the oxide of iron difficult of solution. It is necessary to ensure the complete oxidation of the finely-divided iron, as otherwise it is very difficultly soluble in hydrochloric acid.

In all cases on adding acid to the “ash,” a marked effervescence showed that alkaline carbonate (from the alkali-containing ferrocyanide) was present. Pure blue leaves an “ash” completely soluble in hydrochloric acid, but in the case of adulterated Prussian blue or of diluted blues, barium sulphate or other insoluble matter must be filtered off and weighed before precipitating with ammonia. The iron in the (weighed) mixed oxides was determined by titration with potassium bichromate after reduction with stannous chloride, the alumina being of course estimated by difference. A portion of the filtrate was evaporated to dryness, and, after volatilization of the ammonium salts, the alkaline salts were weighed, and the chlorine therein

* *Loco citato.*

determined by titration with silver nitrate. In another portion of the filtrate the sulphuric acid was determined, and from these data—viz., the weight of the alkaline salts, chlorine, and sulphuric acid—we were enabled to show that in all cases the alkali-metal was almost entirely potassium or sodium, and in no case a mixture of the two metals. Another weighed quantity of the pigment was boiled for a few minutes with aqueous potash, and the resulting precipitate of oxide of iron weighed, after ensuring its complete conversion into Fe_2O_3 . This, multiplied by 0.7, represents the "extra radicle" iron.

The following tables represent the composition of eight samples of blue sold by makers of repute as genuine Prussian blue of good quality :

COMPOSITION OF EIGHT SAMPLES OF COMMERCIAL "PURE" PRUSSIAN BLUE.

	I.	II.	III.	IV.	V.	VI.	VII.	VIII.
Moisture (lost at 100° C.) ...	5.61	3.54	5.36	5.45	74.53	5.32	5.56	5.61
Water of combination, etc. ...	15.46	18.18	6.22	13.07	3.08	7.86	14.60	16.93
* Cyanogen ...	37.72	41.10	42.97	37.90	10.64	39.91	40.19	40.86
† Iron ...	29.48	32.16	34.27	30.32	7.97	30.94	31.94	31.25
Aluminium ...	1.82	.52	—	3.17	.72	1.00	1.43	1.52
Alkali metal (Na) ...	7.60	(K) 4.50	(K) 7.72	(K) 2.25	(K) 1.06	(K) 11.31	(Na) 2.52	.76
Alkaline sulphate ...	2.31	—	3.46	7.84	2.00	3.66	3.76	(Na) 3.07
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
* Nitrogen ...	20.31	22.13	23.14	20.18	5.73	21.49	21.64	22.00
† "Extra radicle" iron	16.66	18.99	19.05	16.04	—	16.21	18.98	19.38

COMPOSITION OF DRY MATTER OF ABOVE SAMPLES (DRIED AT 100° C.).

	I.	II.	III.	IV.	V.	VI.	VII.	VIII.
Water of combination, etc. ...	16.38	18.85	6.58	13.87	12.03	8.35	15.46	17.95
* Cyanogen ...	39.96	42.61	45.41	40.06	41.77	42.14	42.55	43.29
† Iron ...	31.23	33.34	36.21	32.05	31.30	32.67	33.82	33.09
Aluminium ...	1.93	.54	—	3.35	2.90	1.03	1.52	1.61
Alkali metal ...	8.05	4.66	8.16	2.38	4.16	11.94	2.67	0.81
Alkaline sulphate ...	2.45	—	3.64	8.29	7.84	3.87	3.98	3.25
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
* Nitrogen ...	21.52	22.94	24.45	21.57	22.49	22.69	22.91	23.30
† "Extra radicle" iron ...	17.65	19.69	20.02	16.95	—	17.12	20.09	20.51
Intra-radicle iron (found) ...	13.58	13.65	16.19	15.10	—	15.55	13.73	12.78
Intra-radicle iron (calculated from nitrogen) ...	14.34	15.30	16.30	14.38	14.99	15.12	15.28	15.54

In looking at these figures, it will be seen that the percentages of iron and nitrogen do not vary greatly. The amounts of iron in the iron-cyanogen radicle, calculated from the percentage of nitrogen and from the difference between total and extra radicle iron, do not agree closely in all cases. We can offer no explanation of this. Taking the figures for the dry matter, the greatest difference in iron was in the cases of III. and I., 4.98 per cent., and the greatest difference in nitrogen—between III. and I. again—2.93—differences which, calculated on the mean percentages, are under 16 and under 13 per cent. of the totals respectively.

It will also be noticed that seven out of the eight samples contained aluminium. This is not to be considered as an adulterant, as alum is often added to the sulphate

of iron used in precipitating the ferrocyanide without any idea of adulteration, but probably to cause the precipitate to settle better and to dry in a more coherent manner. It probably exists as aluminium ferrocyanide, a compound described by Tissier (*Comp. rend.*, xlv., 232). This has no colouring properties, and, of course, the iron and nitrogen in the iron cyanogen radicle of this compound appear in the total iron and nitrogen as determined. If merely the *ammonia precipitate* is taken and called "ferric oxide," the alumina is included as oxide of iron.

We think that the addition of alum to the precipitating tank is unnecessary, especially as the best sample of blue we have examined contained none.

Alkali metal, as iron-alkali-cyanide, is present in every case. This agrees with the statement of Williamson and others that a blue cannot be obtained free from alkali-metal, except when hydroferrocyanic acid is substituted for alkaline ferrocyanide in the manufacture (the alkaline sulphate present in each case is, of course, due to imperfect washing).

In fact, Prussian blue, if by that term we mean ferric ferrocyanide $\text{Fe}_3(\text{CN})_{18}$, is not known to the commercial world. Commercial Prussian blue is a mixture of Williamson's blue with other iron-alkali cyanides, and often with aluminium-iron cyanides, altogether a most complex substance.

An examination of the watery extract showed, in the cases of I. and VI., the presence of alkaline ferrocyanide. This is rather remarkable, as with the quantities usually taken the iron is in excess. We believe that these came from the same maker, but are sure, however, that both samples were sent out as bona-fide Prussian blue.

The following notes on appearance of samples in lump and in powder, depth of colour, etc., may be of interest :

Condition.	Appearance.	Powdered Samples.	
		Classification in order of Depth of Colour.	Quality of Tint.
I. Lumps, conchoidal fracture ...	blackish copper glance, but not a bright sheen	2	6
II. Lumps	blue, pale-looking, no coppery glance	6	4
III. Lumps, conchoidal fracture ...	rich blue, bright coppery sheen ...	4	1
IV. Lumps, conchoidal fracture ...	ditto, but not as good as III. ...	3	2
V. Paste	dried, with coppery lustre	—	—
VI. Lumps, conchoidal fracture ...	blackish coppery glance, but not a bright sheen	1	7
VII. Powder	—	7	5
VIII. Lumps	light blue, no coppery glance ...	5	3

The mean percentage of iron in the dry matter of these eight samples was 32.96. This would give 3.03 as the factor for calculating iron to Prussian blue. The mean

percentage of nitrogen was 22.73; this would give as a factor for calculating nitrogen to Prussian blue 4.4. The following tables show the accuracy to which these factors gave the percentage of Prussian blue in the eight samples examined:

In Dry Matter.	I.	II.	III.	IV.	V.	VI.	VII.	VIII.
N \times 4.4	94.69	100.93	107.58	94.91	98.95	99.83	100.80	102.52
Fe \times 3.03	94.63	101.02	109.71	97.11	94.84	98.99	102.47	100.26

Of course, these are merely empirical factors, based on the examination of commercial samples of varying composition, and do not possess the accuracy of a factor to reduce one definite chemical compound to terms of another, but, at any rate, taken together they are of sufficient accuracy for calculating the percentage of Prussian blue in pigments containing that colour. The nitrogen alone in most cases would be sufficient.

We feel justified in stating that blue sold as Prussian blue should contain at least 20 per cent. of nitrogen and 30 per cent. of iron calculated on the dry matter, and, after burning, should be entirely dissolved by hydrochloric acid. A "dry" blue should certainly contain under 7 per cent. moisture. Another important point is that the sulphuric acid used in the Kjeldahl nitrogen determination should not be blackened. Pure cyanides dissolve in sulphuric acid without any charring, which, if it occurs, indicates the presence of organic adulterants. A case of this kind will be noted later.

The watery extract should not indicate the presence of excess of alkaline ferrocyanide. We consider that this is evidence of defective manufacture, although it cannot, of course, be regarded as adulteration. Still it—containing as it does iron and nitrogen—is partly included in the analysis as Prussian blue, whereas it has no pigmentary value, and its solubility in water does not add to the weathering qualities of the colour.

COMMERCIAL PIGMENTS.

The following cases of examination of an adulterated sample of Prussian blue (of German manufacture) and of some colours ground in oil possess some interest.

A rather pale-looking sample of dry powdered Prussian blue, which we were told was a cheap sample of German manufacture, gave only 14.35 per cent. of nitrogen. This at once aroused our suspicions, especially as, instead of becoming colourless in a few minutes on boiling with sulphuric acid, a decided blackening occurred. This clearly pointed to the presence of an organic adulterant. On further examination, the following figures were obtained:

Moisture	11.35
Water of combination, organic adulterant, alkalis, etc. ...	37.76
Cyanogen*	26.65
Iron	22.67
Aluminium	1.14
Insoluble	43
	<hr/>
	100.00
Per cent. nitrogen \times 4.4	63.10
„ iron \times 3.03	68.69
* Nitrogen	14.35

This sample not only contained more water than is usual, but was clearly deficient in blue. We think that one is justified in stating that it did not contain more than 70 per cent. of Prussian blue.

Two samples of Brunswick green ground in oil were examined. Brunswick green is a Prussian blue-lead chromate green mixed with barium sulphate ("barytes"). A portion from which the oil had been extracted was gently ignited and treated with hydrochloric acid, the solution of lead, iron, and chromium chlorides filtered from the barium sulphate, and the lead precipitated with sulphuric acid. In the filtrate from the lead sulphate the "ammonia precipitate" containing the mixed oxides of iron and chromium was determined, the oxides were separated by fusion with alkaline carbonate and nitrate and extraction of the fused mass with water, Fe_2O_3 being insoluble. Direct titration with $\text{K}_2\text{Cr}_2\text{O}_7$ was difficult, as the colour of the chromium masked the reduction of the iron with stannous chloride :

				Light-green.		Dark-green.
Oil	11.27	...	13.55
Prussian blue ($\text{Fe} \times 3.03$)	1.01	...	4.21
Chrome yellow	4.98	...	7.10
Barium sulphate	82.74	...	75.14
				100.00		100.00

The nitrogen was determined in each of these samples in about 5 grammes, and was .19 and .72 respectively, which, using 4.4 as a multiplier, would give .83 and 3.16 per cent. of Prussian blue in these samples. We are much more inclined to rely on a direct determination of this kind than on the determination of the iron by the method described. The method of analysis for these colours described by Hurst, and due to Brown (*Chem. News*, December 31, 1886), seems still more unsatisfactory. The green is treated with hydrochloric acid to dissolve out the lead chromate (of course, as lead and chromium chlorides). The residue, barytes and Prussian blue, is ignited to decompose the blue, weighed, treated with aqua regia to dissolve out oxide of iron (*and alkalis*), and the insoluble portion again weighed. The difference is to be taken as oxide of iron and multiplied by 2.212 to give Prussian blue. The lead and chromium are, of course, determined in the original filtrate.

In the case of colours ground in oil, the Kjeldahl method is particularly applicable for the determination of nitrogen, and, as now it is very general to sell these colours in oil instead of dry, we think that the nitrogen determination will be found a very convenient method of ascertaining (approximately) the amount of Prussian blue in the many diluted colours made from it. Adulteration with nitrogenous matter is not probable, and so is not likely to lead to erroneous results. We intend to continue the examination of all samples which come under our notice by this method, which is rapid (we have obtained a result in under one hour), and at once points to any serious sophistication.

In conclusion, we have to mention that this work was done in the laboratory of the London County Council in the course of our regular official duties. We also desire to express our thanks to Messrs. S. Lambert and Co., Sir William Rose and Co., and Messrs. Colthurst and Harding, for providing us with samples and information respecting their manufacture.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD ANALYSIS.

Iodine Number and Refractive Index of Cocoa Butter. A. Strohl. (*Zeit. anal. Chem.*, 1896, xxxv., 166-169.)—In order to determine what were the actual limits in these constants, the author examined samples of all the known trade varieties. The iodine number was determined by Hübl's method, an excess of iodine (over 60 per cent.) always being allowed. The results showed that the iodine number of pure cocoa butter, whatever its source or method of preparation, varied between 32.8 and 41.7.

The refractive index determined at 40° C. varied between 1.4565 and 1.4578, these numbers corresponding to 46 and 47.8 on the scale of the Zeiss butyro-refractometer. A certain parallelism was noted between the iodine number and refractive index, a cocoa butter with a low Hübl number having also less refractive power. (Cf. Hehner, *ANALYST*, xx., 136.) C. A. M.

Testing for Darnel in Flour. A. Deros. (*Ann. de Chim. Analyt.*, i. [10], 190, 191.)—As the colour-reaction with sulphuric acid, described by Wagner and Ginsberg (*Ann. de Chim. Analyt.*, i. [8]), is due to saponin, it cannot be considered as characteristic of darnel alone. Corn-cockle, which is frequently present in wheat, barley, and other flours, also contains saponin. Neither do the properties of the fat extracted by ether, nor the polygonal form of the starch grains (when in small proportion), considered separately, afford convincing proof of the presence of darnel; but when these characteristics are cumulative a positive decision may be given.

C. S.

Chemical Detection of Horseflesh. Courlay and Coremons. (*Ann. de med. veterin. Through Zeit. Nahr. Untersuch.*, 1896, 173, 174.)—The method here described is based on the same principle as that of Brautigam and Edelmann (*ANALYST*, xx., 95 and 252), but is claimed to be more easy of application. About 50 grammes of the substance to be examined in as fresh a state as possible are finely divided and boiled from fifteen to thirty minutes with 200 c.c. of water. After cooling, the broth is filtered, and then tested with a few drops of iodine solution, prepared by dissolving 2 parts of iodine and 4 parts of potassium iodide in 100 parts of water. A brown coloration, disappearing on warming to 80° C. and reappearing on cooling, shows the presence of horseflesh. In the case of the addition of flour (e.g., in sausage) the blue colour may mask the glycogen reaction. This is obviated by adding two or three times the volume of concentrated acetic acid to the broth, filtering, and again testing the filtrate with the iodine solution. No reaction was obtained by the authors with the flesh of cattle, calves, pigs, dogs or cats, but this observation does not apply to the flesh of the foetus of any of these animals. C. A. M.

Horse-flesh and Horse-fat. R. Frühling. (*Zeit., ang. Chem.*, 1896, 352, 353.)
-Not being able to obtain conclusive results with Edelmann's glycogen reaction, the

author has attempted to use the iodine number of the fat as a means of detection. Fat taken from different parts of a freshly-killed horse gave the following values :

	Back.	Heart.	Kidney.	Mean.
Specific gravity at 50° ...	0.8963	0.8948	0.8987	0.8966
" " at 17.5° ...	0.9159	0.9167	0.9212	0.9180
Iodine number (Hübl) ...	79.9	77.4	82.6	80.0
Köttstorfer number ...	182.8	184.7	187.6	185.0
Iodine number of fatty acids ...	81.4	78.3	84.0	81.6
Melting-point of fatty acids ...	52.53°	40.41°	53.54°	48.8°
Solidification - point of fatty acids ...	45.43°	34.32°	48.47°	41.5°

The iodine numbers of the fat extracted from three specimens of sausage containing horseflesh were as follows :

	Iodine number.
Sausage made from pure horseflesh ...	72.5
" consisting of horseflesh, with 15 per cent. of pork ...	62.3
" " " " 50 " "	57.2

Since lard has an iodine number of from 56.9 to 63.8 (according to Benedikt, pork), it is obvious that this method would lead to no certain conclusion. C. A. M.

The Estimation of Caffeine. M. Georges. (*Jour. Pharm. et Chim.*, 1896, xvi, 58, 59.)—The sample (tea, etc.) is finely powdered, and 5 grammes mixed with fine sand, and lixiviated with a 1 per cent. aqueous solution of salicylate of soda until the caffeine is completely extracted. The liquid is concentrated to about 50 c.c. on the water-bath, filtered, and the filter washed with the salicylate solution. The caffeine is then extracted from the solution with chloroform, and the latter evaporated. The caffeine is perfectly white and free from impurities. In a sample of black tea analysed by this method 3.92 per cent. of caffeine was obtained. No comparative figures of the results obtained by other methods are given. Sodium benzoate or sodium cinnamate may be substituted for sodium salicylate, but caffeine is not quite as soluble in their aqueous solutions as in that of sodium salicylate. C. A. M.

The Estimation of Caffeine in Tea. A. Petit and P. Terrat. (*Ann. de Chim. Analyt.*, i. [12], 228.)—The researches made by the authors show that, instead of magnesia or lime being necessary to liberate the caffeine from its state of combination in the tea, as supposed by Commaille, before it can be extracted by chloroform, water alone is sufficient to effect this result; and, further, that it is necessary to perform the extraction on *moist* tea, since the caffeine resumes its insoluble condition during the process of drying. Referring to the method, proposed by Paul, of extraction by alcohol, they find that this solvent requires dilution to 60° or 80° strength, 98° alcohol only extracting 0.88 per cent. of caffeine from a tea containing 2.50 per cent. The maximum was obtained by means of the Commaille method performed with a *moist mixture*, and by the Petit and Legrip method, which latter consists in treating 25 grammes of powdered tea with three times its weight of boiling-water for a quarter of an hour with frequent shaking; then evaporating on the water-bath until the tea just merely moistens the fingers when pressed, whereupon the chloro-

form extraction is performed, and continued until the residue, after being taken up by boiling water and filtered, no longer gives any precipitate or turbidity with a solution of tannin. The chloroform being then distilled off, the extract is taken up by boiling water, filtered through a moist filter, carefully washed, evaporated on the water-bath, and weighed. If the crude caffeine contains chlorophyll, it should be treated by Grandval and Lajoux's method—viz., dissolving in 15 c.c. of 10 per cent. sulphuric acid in the cold, filtering, neutralizing the acid by ammonia, and evaporating to complete dryness, taking up the residue with chloroform and evaporating this solution at a low temperature.

In all the methods tested, wherein the extraction by chloroform or alcohol (except dilute alcohol) was performed on dry tea, whether with or without the presence of lime or magnesia, the amount of caffeine obtained was far below the truth, and although the method of Grandval and Lajoux—who substituted ammonia for the other alkaline bases mentioned, and employed ether in the formation of the mixture—gave nearly as good results as that of Petit and Legrip, described above, the latter is considered to be preferable as being simpler. The alcohol methods take more time, and the purification of the caffeine is more difficult than by the chloroform method.

C. S.

On the Estimation of the essential Oil of Mustard in Feeding Stuffs. M. Passon. (*Zeit. angewan. Chem.*, 1896, 422, 423.)—The process here described depends on the fact that mustard-oil is soluble in glacial acetic acid, and, as the author's experiments prove, may be quantitatively determined in such solution by Kjeldahl's method. The apparatus required consists of a flask, *a*, fitted with a safety tube and a tube delivering into a second flask, *b*, from which passes a tube into a small beaker, *c*, containing 20 c.c. of sulphuric acid, to absorb any mustard-oil escaping from *b*. The flask *b* contains from 25 to 75 c.c. of glacial acetic acid and a little zinc and iron dust to partially reduce the mustard-oil to allylamine. About 25 grammes of the oil-cake are mixed with 300 c.c. of water and 0.5 gramme of tartaric acid in flask *a*, and slowly distilled for about two hours. The contents of *b* and *c* are then mixed in a Kjeldahl flask, and the nitrogen determined in the usual way. One c.c. of decinormal alkali corresponds to 0.0099 gramme of allyl iso-thiocyanate (C_3H_5NCS). In a sample of rape-seed cake the author found (1) 0.205 per cent. and (2) 0.219 per cent. of mustard-oil. In the second case the apparatus was allowed to stand for twenty-four hours, and the zinc and iron dust not added until immediately before the distillation.

C. A. M.

Application of the Röntgen Rays to the Analysis of Vegetable Substances. F. Ranwez. (*Bull. de l'Ass. belge*, 1896, x., 44-48.)—The author describes how, by means of photographs taken with the Röntgen rays, adulteration of vegetable substances with mineral matter may be readily detected. So far his experiments have been confined to the examination of samples of saffron, some of which were pure, and others adulterated with barium sulphate.

C. A. M.

Detection of Egg-yolk in Pastry, etc. E. Spaeth. (*Zeit. Nahr. Untersuch.*, 1896, x., 171-173.)—In order to determine whether flour products have been coloured

with yolk of egg or with other substances such as saffron or picric acid, the author examines the fat. According to Gobley and others, egg-yolk has the following percentage composition :

Water	5.18	Cholesterin	0.4
Vitellin	15.8	Glycerin-phosphoric acid	1.2
Nuclein	1.5	Lecithin	7.2
Palmitin }	20.3	Cerebrin	0.3
Olein }		Colouring matters	0.5
Stearin }		Salts	1.0

Of these constituents, triolein, tristearin, tripalmitin, cholesterin, and glycerin-phosphoric acid are soluble in ether.

In the subjoined table the characteristics of the fat are given, together with those of the fat from wheat flour :

	Egg-yolk Fat.	Wheat-meal Fat.
Specific gravity at 100° C. (water at 15° = 1) ...	0.881	0.9068
Melting-point of fatty acids ...	36°	34°
Saponification number (mean of 3 results) ...	184.43	166.5
Iodine number (mean of 3 results) ...	68.48	101.5
Iodine number of fatty acids ...	72.6	—
Reichert-Meißl value ...	0.66	2.8
Refractive index at 25° C. ...	1.4713	1.4851
„ „ on Zeiss refractometer scale ...	68.5	92.0

Although a quantitative determination is not possible, yet by means of the fat an approximate judgment as to the amount of egg-yolk present can be formed. Flour products with only a slight addition of yellow of egg invariably give more ether extract than those derived from wheat meal and coloured. For the examination from 100 to 200 grammes of the flour products are ground as finely as possible and extracted with ether in a Soxhlet's extractor. The ether is evaporated, and the residue taken up with petroleum spirit of low boiling-point. After standing several hours until no more solid matter separates, the liquid is filtered, the petroleum spirit evaporated, the residue dried in the water-oven, and its iodine number and refractive index determined. When the iodine number exceeds 98, and the phosphoric acid in the fat is below 0.005 per cent., there cannot be more than traces of egg-yolk. Of course, it must not be lost sight of that wheat-flour itself also contains phosphoric acid.

C. A. M.

Estimation of Free Tartaric Acid in Wines. L. Magnier de la Source. (*Ann. de Chim. Analyt.*, i. [11], 205.)—In order to ascertain whether the action of potassium acetate in preventing the precipitation of potassium bitartrate in a mixture of ether and alcohol is transitory or permanent, 60 c.c. of a wine, to which 13.5 grammes tartaric acid per litre had been added, were divided into three equal parts, to each of which were added 100 c.c. of ether-alcohol mixture, and, in addition to this, to the first (a), 5 c.c. of distilled water, to the second (b), 5 c.c. of concentrated potassium acetate, and to the third (c), 5 c.c. of a concentrated solution of potassium bromide. After standing for two days at a temperature approaching zero, the tartaric

acid was estimated, and found to be, in *a*, 6.96 grammes, in *b*, 11.60, and in *c*, 19.20 grammes. The amount retained in solution by the potassium acetate in *b* was therefore 12.24 grammes, practically the whole of which can be precipitated in 48 hours by means of potassium bromide. Of the 19.20 grammes found in *c*, 2.50 grammes were present originally in the wine, leaving 16.70 grammes corresponding to 13.30 grammes of the 13.50 grammes of tartaric acid added, whereas only about 50 per cent. of the tartaric acid is precipitated from *b*. If the filtrate from *b* be united with the washings and an excess of acetic acid added to one moiety, crystals of tartar will develop in the course of 24 hours, whilst the other moiety will not show any trace of deposit.

These results show the defects of potassium acetate as a precipitant of tartaric acid, and at the same time demonstrate the exactness of the results obtained by potassium bromide (or chloride).
C. S.

Composition of Goose-fat. J. Rozsenyi. (*Ber. Chem. Inst., Buda Pest*; through *Zeit. ang. Chem.*, 1896, 364.)—The following constants were obtained with four samples of goose-fat rendered by the author:

	1.	2.	3.	4.
Specific gravity (15°) ...	0.9229	0.9258	0.9228	0.9300
Melting-point { commenced ...	26.6°	30.0°	29.5°	29.5°
{ ended ...	27.5°	31.4°	31.7°	31.0°
Solidification-point ...	18.4°	18.3°	18.1°	18.1°
Melting-point, fatty acids (commenced) ...	35.2°	35.3°	39.0°	—
Melting-point, fatty acids (ended) ...	36.6°	37.5°	40.2°	—
Iodine number ...	58.7	62.8	66.4	62.5
Reichert-Meißl number ...	0.3	0.3	0.3	0.2
Köttstorfer number ...	193.0	191.2	191.6	193.0
Hehner number ...	94.5	95.3	95.1	—
Refractive index at 40° (Zeiss) ...	50.5	50.0	50.5	50.5

C. A. M.

The Amount of Copper in Vegetable Produce. V. Vedrödi. (*Chem. Zeit.*, xx., 1896, 399.)—Considerable differences in opinion have arisen in the past between the author and other observers regarding this question, and Lehmann in particular has recorded a number of figures showing only about the one-hundredth part of copper found by the present investigator. Lehmann's process was a colorimetric one, the red colour produced by ferrocyanides being employed to determine 0.02 to 0.2 milligramme of copper, and the blue yielded by ammonia when as much as 0.2 to 1 milligramme was present. The samples were burnt in a porcelain crucible after addition of sulphuric acid, the ash dissolved in acid, the solution evaporated, and the ammonium salts filtered off. Vedrödi has always objected to the use of the acid, and prefers to incinerate the substance alone in a muffle, dissolve the ash in hydrochloric acid, pass sulphuretted hydrogen, filter, wash and ignite the precipitate, again dissolve and throw it down as before, finally weighing and calculating it into copper oxide. Finding that he originally made a slight mistake in the allowance for the weight of

the filter ash, he has checked the process on minute quantities of pure metal, and has made a number of fresh determinations, the results of which are recorded in the table. He remarks that the form in which the copper exists in these substances is unknown; and he appears to doubt the toxicity of some of the organic salts, pointing out that in many places pickled gherkins, etc., are habitually coloured with copper, and sold without any complaints arising about evil effects produced by their consumption.

MILLIGRAMMES OF CU PER KILO.

1894.				1895.			
		Min.	Max.		Min.	Max.	
Winter wheat	...	80	710		200	680	
Summer wheat	...	190	630		190	230	
Rye	...	60	90		10	30	
Barley	...	80	120		10	70	
Oats	...	40	190		40	200	
Buckwheat	...	160	640		150	160	
Haricots	...	160	320		110	150	
Lentils	...	120	150		110	150	
Peas	...	60	100		60	110	
Soja beans	...	70	100		70	80	
Lupins	...	80	190		70	290	
Mustard-seed	...	70	130		60	70	
Capsicum...	...	790	1350		230	400	

F. H. L.

Notes on Cubebs. K. Peinemann. (*Arch. Pharm.*, 1896, ccxxxiv., 241; through *Chem. Zeit. Rep.*, 1896, 162.)—Cubebin exists chiefly in the fruit of the cubeb plant, but it may also be found in the stalks, and to a lesser extent in other portions of the plant. It is present in the pericarp as well as in the perisperm, as is also the case with piperine in the ordinary pepper. With the exception of Lowong pepper, those peppers in which cubebin or its derivatives are to be detected never contain any alkaloid (piperine), and the converse of the statement also holds good.

Cubebin contains $C_{20}H_{20}O_8$; it crystallizes in needles, melting at $125^\circ C$. With strong sulphuric acid it gives a fine purplish violet colour. Dissolved in chloroform, it rotates light to the left, pseudo-cubebin to the right.

F. H. L.

TOXICOLOGICAL ANALYSIS.

Purification of Arsenical Sulphuretted Hydrogen by Iodine. Schlagdenhauffen. (*Bulletin Commercial*, April, 1895 (?), p. 178; through *Ann. de Chim. Analyt.*, i [11], 209.)—Jacobson and Brunn (*Berichte*, xx., p. 1099; xxi., p. 2546) remove the arseniuretted hydrogen present in sulphuretted hydrogen (prepared from commercial hydrochloric acid and sulphide of iron) by passing the gas over iodine, the iodide of arsenic formed collecting in the U-tube of the apparatus. If, however, the sulphuretted hydrogen is liberated too quickly, the action of the iodine is incomplete,

and besides, the investigations of Skraup, confirmed by those of the author, show that the arsenic is not all liberated in the early stages of the primary reaction, as was asserted by Otto, but continues to come off throughout, wherefore it is necessary to pass the *whole* of the gas—and not merely the first portions—over the iodine. The hydriodic acid formed simultaneously with the iodide of arsenic must be removed from the sulphuretted hydrogen by absorption in water, otherwise, in cases where the sulphuretted hydrogen is allowed to react on metals or alloys contaminated with arsenic, the iodine will be present in the ammoniacal solution submitted to the Marsh test, and, being converted into iodic acid by the action of the nitric acid employed in the test, will monopolise the first portions of the nascent hydrogen formed, and so retard the deposition of the arsenical ring, thereby leading to the conclusion—unless the duration of the test is prolonged—that there is no arsenic present.

The author prefers to employ so-called pure hydrochloric acid instead of the commercial acid which often contains considerable quantities of arsenic. C. S.

Detection of Mercury in Cases of Poisoning. D. Vitali. (*Chem. Zeit.*, xx., 1896, 517.)—The organic portion of the substance under examination is destroyed according to Fresenius and Babo's process, and the filtered liquid evaporated as far as possible without causing the mercuric chloride to crystallize out. It is then treated with a stream of sulphuretted hydrogen for several hours, being kept warm all the time. The sulphide is washed by decantation, dissolved on the water-bath in aqua regia, and the chlorine and nitric acid removed by evaporation with hydrochloric acid. The solution is introduced into a porcelain basin, in which are placed a piece of goldfoil and an iron nail. In an hour's time the whole of the mercury will probably be deposited partly on the gold and partly on the nail. These are taken out, washed and dried, put into a small test-tube, and ignited gently. The mercury yields the characteristic gray sublimate, which may be identified with greater certainty by adding a minute crystal of iodine, and heating gently once more; if an excess of iodine is used, the yellow or red colour of the iodide may be masked, but on warming in a current of air it will be seen distinctly. The reaction detects 0.01 milligramme of mercury.

The iron and gold may also be heated on the water-bath in a basin covered with another, the outside of which is moistened with gold chloride, when the gold is reduced to the metallic state. F. H. L.

ORGANIC ANALYSIS.

Action of Nascent Bromine on Naphthols and Naphthylamine. W. Vaubel. (*Zeit. anal. Chem.*, 1896, xxxv., 164-166.)—With a view to subsequent application to analytical processes, the author has studied the action of nascent bromine liberated by adding hydrochloric acid to a solution of potassium bromide and bromate, on naphthol and naphthylamine derivatives dissolved in acetic acid.

a-Naphthol.—Absorbs two atoms of bromine rapidly. Subsequently a further

absorption slowly occurs, which renders the end reaction obscure. Consequently a bromination method for estimating the amount of this substance is not practicable.

Ethyl Ester of α -Naphthol.—Absorbs one atom of bromine, forming the brom-naphthol ethyl ester. Melting-point, 48° C. End reaction distinct.

β -Naphthol.—Absorbs one atom. End reaction well marked.

Methyl Ester of β -Naphthol.—Absorbs one atom. End reaction clear. The bromide separates in white shining needles. Melting-point, 85° C.

Ethyl Ester of β -Naphthol.—Behaves like the methyl compound, but the absorption is slower. The mono-bromide separates in plates, melting at 54° C.

α -Naphthylamine.—In the main absorbs two atoms of bromine. Owing to the simultaneous formation of small quantities of oxidation products, bromination cannot be employed as a method of estimation.

α -Acetnaphthylamine.—Absorbs one atom. End reaction well marked. The mono-bromide melts at 190° C.

β -Naphthylamine.—Rapidly absorbs two atoms. End reaction not clear, owing to formation of oxidation products.

β -Acetnaphthylamine.—One atom absorbed. End reaction clear. Compound melts at 140° C. C. A. M.

Adulteration of Rape-seed. Pajot. (*Ann. de Chim. Analyt.*, i. [10], 187-190).

—The author having been called upon to report on the percentages of oil in certain oleaginous seeds, discovered a somewhat extensive adulteration of German rape-seed by two varieties of mustard, viz., charlock and *Brassica juncea*, coloured to resemble rape. The blue colouring matter is easily extracted by cold water in about fifteen to thirty minutes, or much more readily by the action of an acid, in which case a rose-coloured solution, turning blue on the addition of an alkali, is obtained. The test may also be performed by placing the seed on a white filter-paper moistened with acid or water, the colour appearing at the points of contact. The aqueous extract is found to contain salts of iron and sulphates, and has an alkaline reaction, whereas pure rape-seed extract is acid, and contains neither iron nor sulphates. The colouring matter is believed to be a product analogous to litmus, and, being applied when the seeds are newly harvested, is fixed and modified by the integument of the seed. A considerable profit must be derived from the fraud, since the charlock is only worth about three shillings per cwt. and the *Brassica juncea*-seeds about six shillings, whereas rape-seed averages about nine shillings per cwt. The yield of oil is from 10 to 15 per cent. less than from pure rape-seed, and its quality inferior. In addition to this, the oil-cake is rendered unfit for cattle food, and is only suitable for manure. C. S.

Estimation of Glucose. Causse. (*Journ. de Pharmacie*, 1896, p. 433; through *Ann. de Chim. Analyt.*, i. [10], 192.)—The author criticises the method proposed by Gerrard (*Pharmaceutical Journ.*, April, 1895), and considers that his own process (*Journ. de Pharm.*, xix., 171) is preferable, since the potassium ferrocyanide employed to prevent the precipitation of the cuprous oxide has no action on Fehling's solution.

Good results are obtained by the author's method in the detection of sugar in urine and blood. C. S.

On the Distillation of the Lower Acids of the Fatty Series in Presence of Water. E. Sorel. (*Ann. de Chim. Analyt.*, i. [13], 245.)—The difference between the proportions found by Duclaux and Muntz respectively in their researches on this subject, is due to the conditions under which the experiments were made—i.e., to the modifications produced by condensation in the composition of the distillates prepared in retorts exposed to radiation. To ensure regularity of results, the retorts should be surrounded by a metallic jacket, heated by a ring-burner to a temperature exceeding the boiling-point of the still contents before distillation is commenced; and, furthermore, the neck of the retort should be bent so as to prevent the return of any condensation products to the still.

The basis of the calculation of the ratio of the acids in the fractions of distillate to the amounts in the liquid under examination is determined by distilling 550 c.c. of a liquid of known composition, dividing the distillate into nine portions of 50 c.c.—disregarding the first 50 c.c. as containing the products passing off below the boiling-point—and leaving the tenth portion of the liquid in the still.

The purity of the acids is checked by the preparation of their compounds with barium.

From the table prepared by the author, it is deduced that the Duclaux method—with different co-efficients—is applicable to formic acid in the presence of acetic acid, provided they do not together constitute more than 10 per cent. of the original liquid; but in the case of the higher acids the method is not conclusive, particularly as the exact conditions of Duclaux' and Muntz' methods of working are unknown

C. S.

The Valuation of Glue. C. Stelling. (*Chem. Zeit.*, xx., 1896, 461.)—As the value of glue depends simply on its adhesive power, the only reasonable method of testing it is to attempt to determine the amount of substances other than gelatin ("non-glues") it contains. These bodies react with tannin in a manner similar to glue itself; while the quantity of water the sample will absorb depends chiefly on the process and temperature of manufacture. The following method gives comparative results, and at least indicates the source of the product, whence some idea can be obtained as to its probable value. 15 grammes are soaked over night in 60 c.c. of water in a 250 c.c. flask. Next day the jelly is dissolved on the water-bath, and the loss by evaporation made up. The flask is then filled with 96 per cent. spirit, and the whole thoroughly shaken. After standing 6 hours, 25 or 50 c.c. of the liquid are filtered off, the residue consisting of the non-glues dried at 100° and weighed. Five different sorts of gelatin gave 2.53 to 4.53 (mean 3.39) per cent. of non-glues; five glues gave 2.0 to 4.70 (mean 3.49); three leather glues 4.30 to 7.60 (5.73); four bone glues from acid bones 9.24 to 11.84 (10.33); three other specimens of the same 13.16 to 16.78 (15.15); seventeen pressed glues from neutral bone powder 14.30 to 32.10 (20.66); one of whale glue 22.0; and two of a material used for the clarification of wine 33.20 and 59.30 per cent. of non-glues respectively.

F. H. L.

Estimation of the "Weighting" Materials in Silk. H. Silbermann. (*Chem. Zeit.*, xx., 1896, 472.)—In the case of white or pale coloured silks, whether in skein or woven, the soluble weighting matters are extracted by boiling with water. The sugars are estimated after inversion by Fehling's solution, and the magnesium salts and the sodium sulphate by sodium phosphate and barium chloride respectively. The residual silk is burnt, and the ash dissolved in hot, fairly strong hydrochloric acid. If tin only be present, as may be told by the behaviour of the acid liquid with sulphuretted hydrogen, the weight of the ash may be multiplied by 1.13 to give the amount of stannic acid originally in the fibre. To calculate how much weighting substance (E) is present in 100 parts of the material, the following formula may be employed: where a = the original weight of the sample, b = the weight after extraction with water, p = the tin oxide ash, and Δ the loss in weight during boiling of the fibre itself, being 20 to 25 for Cuit (Boiled silk), 5 to 9 for Souple (Softened silk), and 0 to 2 for Ecrú:—

$$E = \left[\frac{a(100 - \Delta)}{b - 1.13p} - 100 \right] \text{ per cent.}$$

The tin may also be determined by igniting the sample in a porcelain crucible with an equal weight of caustic soda and a little potassium nitrate. The melt is extracted with water, the solution acidified and the tin thrown down by H_2S . Another sample is boiled in dilute HCl to dissolve the lakes of tin and other metals; if the solution, treated with sodium acetate in excess and a ferric salt, yields a blue black precipitate, the estimation of the individual ingredients is conducted as follows. In the aqueous extract the tannins are precipitated with gelatin; the sugars determined in the filtrate, and the tannins themselves in the deposit. The sample is boiled with dilute HCl and NaHO, and the tannins are precipitated from both liquids. Another portion of the material is burnt to ash, and if the latter is not completely soluble in acid, it contains barium sulphate or silica, or both, as may be ascertained in the usual manner.

Dark silks may also contain iron and chromium salts, as is shown by the brown or green colour of the ash. This should be fused with NaHO and KNO_3 , the melt dissolved in water, which leaves the iron oxide behind, the solution acidified, and examined for the other metals.

Black silks are boiled alternately for half an hour in NaHO (20 grammes per litre) and HCl (250 grammes per litre) four times in each liquid, the fibre being thoroughly washed with hot water between each treatment, and finally cleaned by boiling in soap. If P = the original weight, and D the weight after boiling, the amount of weighting (E) is as follows:—

$$E = \frac{(100 - \Delta)(P - D)}{D}$$

In this instance the value of Δ is 25 for Cuit, 8 for Souple, 0 for Ecrú, and 10 for Fantasia. It is still possible for the fibre to retain metallic salts, and if this be the case it should be burnt to ash. Calling the weight of the latter A, the weight of the

oxides in the fibre averages 1.25A, and therefore the true fibre in P is $D - 1.25A$. The weighting matters in 100 of the original material are

$$E = \frac{(P - D + 1.25A)(100 - \Delta)}{D - 1.25A}.$$

(As an example, $P = 80$ grammes, $D = 62$, and $A = 1.6$; therefore $E = 25$ per cent. in the material; while it contains $80 - 62 + 2 = 20$ grammes of weighting to 60 of true fibre, or 33 per cent. of foreign constituents.)

An alternative process of estimating the true silk fibre in the material consists in a nitrogen determination. The nitrogenous substances, such as tannin, gelatin, and Prussian blue, are first removed by boiling in acid and alkaline carbonate, employing, in the case of Souple and Ecrû, the ammonium salt instead of sodium carbonate. Thus treated, the fibre contains nothing but metallic oxides, and as the amount of nitrogen (17.6 per cent.) in silk is accurately known, the weight of the charge can be readily discovered. Either a combustion with soda-lime or Kjeldahl's process may be employed. The sample must be thoroughly dried, 2 grammes weighed off, and separated into distinct fibres with great care, in order to avoid the production of dust. With this object Persoz has suggested that it should be moistened in 1:3 HCl and then dried and powdered. If a is the weight of the dry sample, and b the amount of nitrogen found,

$$E = \left[\frac{0.176a(100 - \Delta)}{b} - 100 \right] \text{ per cent.}$$

E being, as before, the amount of weighting substance in 100 of the material examined. With suitable precautions this process gives very accurate results. F. H. L.

Saturation Temperatures and Critical Temperatures. Application to General Analysis. L. Crismer and J. Motteu. (*Bull. de l'Ass. belge Chim.*, 1896, ix., 359-375.)—1. *Estimation of Nitro-glycerin in Dynamite.*—Several minor modifications in the process of determining the critical temperature of substances have been adopted (ANALYST, xx., 209, 257). When the temperature does not exceed 170° to 180° C., a glycerin-bath is preferable, and when the temperature is slightly lower, the platinum binding wire may then be replaced by an indiarubber band. Slightly larger tubes (diameter, 6 to 8 m.m. instead of 5 m.m.) are now used. The critical temperature of nitro-glycerin varies with the acidity, but with samples washed until completely neutral it is 46.5° to 46.7° . In order to determine the relation in weight or volume between the two bodies giving the critical temperature of dissolution, it is necessary to plot a curve of the temperatures of saturation. This is done by warming weighed quantities of the substances in small sealed tubes until clear, and noting the temperature at which they become turbid when allowed to cool in the bath. The saturation temperatures of nitro-glycerin in 90 per cent. alcohol are given in the following table, and are also plotted in a curve in the original paper:

Nitro-glycerin. Grammes.	Alcohol, 90 Per Cent. Grammes.	Nitro-glycerin. Per Cent. by Weight.	Alcohol. Per Cent. by Weight.	Temperature of Saturation.	
0.164	1 c.c.	0.818	16.7	83.3	0.4
0.186	"	0.8165	18.58	81.45	7.8 7.8
0.6052	3 c.c.	2.4896	19.55	80.45	11.4 11.4
0.6702	"	2.4928	21.19	78.81	15.4 15.4
0.232	1 c.c.	0.817	22.11	77.9	18 18
0.2405	"	0.812	22.85	77.15	19.8 19.8
0.2555	"	0.820	23.75	76.25	21.6 21.7
0.2595	"	0.8135	24.18	75.82	22.6 22.6
0.2685	"	0.817	24.73	75.27	23.9 23.9
0.3135	"	0.818	27.71	72.29	29.6 29.6
0.4015	"	0.819	32.9	67.1	36.9 36.9
0.460	"	0.8175	36.01	64	40.5 40.5
0.521	"	0.811	39.11	60.9	43.1 43.1
0.576	"	0.8185	41.3	58.7	44.75 44.75
0.6425	"	0.821	43.9	56.1	46.5 46.5

If x grammes of nitro-glycerin have been dissolved in 5 grammes of 90 per cent. alcohol, and the temperature found is 20.1° , this would correspond to 23 per cent. of nitro-glycerin and 77 per cent. of alcohol, and the amount of nitro-glycerin can then be determined by proportion :

$$23 : 77 = x : 5, \text{ or } x = \frac{23 \times 5}{77}.$$

In estimating the amount of nitro-glycerin in dynamite, about 1 gramme of the latter is weighed into a small stoppered flask, together with 3 c.c. of 90 per cent. alcohol, about 2.5 grammes. The flask is then warmed for a few seconds at 30° to 35° , well shaken, the liquid filtered through a dry filter, a small quantity sealed in a tube, and the saturation temperature determined as described above. The tabulated results obtained with dynamites prepared in the laboratory agree well with theory. Two sources of error are possible in this method : either the nitro-glycerin may not have the normal critical temperature, or the dynamite may contain a small amount of moisture. In the former case a preliminary extraction of nitro-glycerin may be made with ether, the critical temperature determined after evaporation of the ether, and a fresh curve plotted on this basis. The latter error may be practically eliminated by increasing the amount of alcohol, but in such a manner that the temperatures of saturation still lie between 15° and 25° .

2. *Estimation of Water in Butter.*—The following example illustrates this application of the process. The butter-fat gave a critical temperature 98° , with 91 per cent. alcohol :

Amount of butter taken	0.7805 gramme.
Water by evaporation	0.1245 = 13.75 per cent.
Alcohol containing 1.344 per cent. of water	1.219	gramme	or 1.5 c.c.	
Critical temperature	106.5 grammes.

This temperature corresponds to an alcohol containing 10.5 per cent. of water, while the alcohol used contained 1.2028 gramme of alcohol and 0.01638 gramme of water. By the proportion $89.5 : 10.5 = 1.2028 : x$ the quantity of water in the alcohol brought

to 10.5 per cent. = 1.1411 grammes, of which 0.01638 gramme was present in the original alcohol. Therefore the quantity of water derived from the butter = 0.1247 gramme, or 13.77 per cent.

The method gives exact results when the butters are not salted, but when there is any notable amount of salt present the figures are too high.

3. *Estimation of Sulphur in Gunpowder.*—The method is analogous to that employed for dynamite, but pure benzene is used instead of alcohol. The saturation temperatures obtained in a series of determinations are represented by a curve, as in the case of nitro-glycerin. A curve is also plotted for the temperatures given by a mixture of alcohol and benzene with sulphur, the latter proceeding being adopted to prevent the crystallization of the sulphur.

4. *Critical Temperatures of Industrial Oils.*—The figures obtained with alcohol (specific gravity 0.8195) were as follows: Mineral oils (various), 135.5° to 140°; valve oil, 197°; animal oil, 120°; sheeps' foot oil, 102°; lard oil, 104°; neats' foot oil, 95°; colza, 132° to 135°; Japanese fish oil, 108° C.

As in the case of nitro-glycerin, the acidity of a fat lowers its critical temperature.
C. A. M.

INORGANIC ANALYSIS.

Examination of Hydrofluoric Acid. K. F. Stahl. (*Zeit. angew. Chem.*, 1896, 225-229).—The impurities always present in the commercial acid are hydrofluosilicic acid, derived from the silica in the fluor spar, and sulphuric acid. These impurities influence the specific gravity of the hydrofluoric acid considerably, so that this constant does not alone give reliable information as to the purity of a sample of unknown origin.

The method of analysis which the author has found most satisfactory is as follows: The sample contained in a lead cylinder is placed in water at 50° C., and the specific gravity determined by means of a platinum hydrometer; then, with a small platinum tube to serve as a pipette, three separate portions are weighed out:

1. Two grammes into a very small platinum crucible (holding about 5 c.c.).
2. " " " large " " (holding about 40 c.c.).
3. Four grammes into a small platinum dish.

A. *Total Acidity.*—The small crucible covered with a lid is placed in a platinum dish, into which is run from 25 to 50 c.c. of normal caustic soda according to the expected percentage of acid, the crucible is overturned, phenol-phthalein added, the acid and alkali mixed, and more alkali added, until after heating to about 50° C. a permanent red colour remains. The number of c.c. used = *a*.

B. *Hydrofluosilicic Acid.*—To the acid in the large crucible about 5 c.c. of water are added, then slowly about 2 grammes of potassium carbonate, followed by 15 c.c. of 50 per cent. alcohol, and as many c.c. of 95 per cent. alcohol as of water used, which will bring the whole to a volume of about 25 c.c. containing about 50 per cent. of alcohol. After standing for an hour the gelatinous precipitate of potassium silicofluoride is filtered off and well washed with 50 per cent. alcohol. It is then placed, together with the filter paper, in a platinum dish with about 25 c.c. of water, warmed

to 50° C., and titrated with normal soda as in the determination of the total acidity. The number of c.c. used = b .

C. Sulphuric Acid.—The acid in the platinum dish is evaporated under a hood until no more acid fumes are expelled, and the syrupy liquid remaining is titrated with normal soda solution, either phenol-phthalein or litmus being used as indicator. The number of c.c. used = c .

The number of c.c. (= a) used in the first titration represents the alkali required to neutralize the hydrofluoric acid, hydrofluosilicic acid, and sulphuric acid.

The number of c.c. used for the hydrofluoric acid alone = a , less the number used for hydrofluosilicic acid and sulphuric acid. But since in the potassium silicofluoride two atoms of fluoride were already neutralized, it would only have required $b + \frac{b}{2}$ c.c. to neutralize the free acid. And as 4 grammes of the substance were used for the estimation of sulphuric acid, the number of c.c. there used must be divided by 2.

Therefore the number of c.c. used for the free hydrofluoric acid is $a - \left(\frac{3}{2}b + \frac{c}{2}\right)$, and since each c.c. normal alkali = 0.020 gramme of HF, and two grammes of the substance were used, this formula also expresses the percentage of free hydrofluoric acid. The percentage of hydrofluosilicic acid is given by the formula $b \times 1.8$, and that of free sulphuric acid by $c \times 1.2$. Other acids, like nitric or hydrochloric acid, are not likely to occur in commercial hydrofluoric acid, and if so, would be readily detected.

The following results were obtained with samples of hydrofluoric acid from various makers. The amounts of the constituents are in percentages.

	1.	2.	3.	4.	5.	6.	7.	8.	9.
Specific gravity		1.299	1.264	1.253	1.244	1.264	1.282	1.247	1.234
Hydrofluoric acid	39.6	42.2	44.3	48.1	48.6	51.1	54.2	48.6	33.5
Hydrofluosilicic acid	2.7	14.9	10.1	4.7	5.0	6.8	8.1	6.3	10.6
Free sulphuric acid		0.8	0.8	4.0	1.9	1.4	1.2	0.8	1.6

C. A. M.

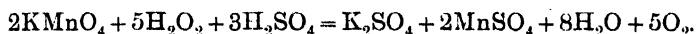
On the Precipitation of Calcium Oxalate. A. Lemoine. (*Bull. de l'Ass. belge*, 1896, x., 124-136.)—To obviate the difficulty of filtering calcium oxalate precipitates which is often experienced, even when the precipitation has been made in boiling solution, the author describes the following method: A sufficient quantity of oxalic acid (preferably solid) is added to the calcium solution, followed by hydrochloric acid until the liquid becomes clear. The solution is boiled and then neutralized with ammonia, avoiding a large excess. The boiling is continued for about ten minutes, or until the greater part of the ammonia has been expelled. The precipitate obtained under these conditions is flocculent, subsides rapidly, and is easily filtered. It may be washed with hot or cold water. It is claimed that in the majority of cases, even when the amount of calcium exceeds 0.5 gramme, less than fifteen minutes is required for precipitation, filtration, and washing, whilst the comparative experiments described in the paper show that it is quite as accurate as the ordinary method. C. A. M.

Resazurine as an Indicator. L. Crismer. (*Bull. de l'Ass. belge*, 1896, x., 22-25.)—This substance gives an intense blue colour with alkalis, alkaline carbonates, and ammonia, whilst acids redden it. It is more sensitive than turmeric, its solutions are stable, and it can be used with borax solution. A decinormal solution of borax, when titrated with the use of this indicator, exactly neutralized a decinormal solution of oxalic acid. On the other hand, it cannot be used for the titration of monobasic organic acids, is less sensitive to carbonic acid than phenol-phthalein, and is uncertain with nitric acid. Treated in alkaline solution with reducing agents (glucose, etc.), it gives a rose colour, with a vermilion-coloured fluorescence.

It is prepared by dissolving 10 grammes of resorcin in 500 c.c. of ether, cooling to 5° C., and adding 8 grammes of fuming nitric acid diluted with ether. After several days, the crystals are collected, washed, and dried. For use as an indicator, 0.20 gramme is dissolved in 40 c.c. of decinormal ammonia and the solution made up to one litre with water.

C. A. M.

A Method for the Standardization of Potassium Permanganate and Sulphuric Acid. H. N. Morse and A. D. Chambers. (*Amer. Chem. Jour.*, 1896, xviii., 236-238.)—To obtain the potassium permanganate of normal composition the solution is prepared from the recrystallized salt, and the suspended manganese oxides removed by filtration. The solution is standardized by adding to a measured quantity of standard sulphuric acid a little hydrogen peroxide, and running in the permanganate so long as the colour disappears. More hydrogen peroxide is then added, and the titration continued to the same point. This is repeated until about 50 c.c. of the permanganate have been reduced, leaving in the solution a minute excess of hydrogen peroxide. Finally, the acid remaining free is titrated with decinormal ammonia, litmus being used as indicator. The quantity of permanganate in 50 c.c. of the solution is calculated from the formula :



In the test experiments described by the authors, 50 c.c. contained 0.1626 gramme, whilst the amounts found by titration with potassium tetroxalate and oxalic acid were 0.1626 and 0.1628 gramme respectively. Sulphuric acid is standardized in the same manner with permanganate solution of known strength. In test experiments, using a solution containing in 50 c.c. 0.17435 gramme of potassium permanganate, the results were identical with those obtained by precipitation as barium sulphate. The hydrogen peroxide is the ordinary commercial solution, first rendered neutral by agitation with zinc oxide which has been heated in a muffle and then filtered through asbestos.

C. A. M.

Estimation of Potash. Ch. Fabre. (*Comptes rendus*, cxxii. [23], 1331.)—The author proposes to expedite the estimation of potash by the platino-chloride method in the following manner :

The potash solution is evaporated on the water-bath with a slight excess of platinic chloride, any ammonia compounds being destroyed by a few drops of nitro-hydrochloric acid. The residue is taken up by a little water and re-evaporated,

then triturated with a little 90° alcohol, collected on a filter, and washed with alcohol till the washings are colorless, whereupon the alcohol in the residue is displaced by ether, which is itself driven off by evaporation, and the residue taken up by boiling water. The next step is to throw down the metallic platinum by means of magnesium, powdered and mixed with alcohol and water, and added gradually, in order that the reaction may not become too violent. 60° is the most favourable temperature, and the formation of oxychloride of magnesium is prevented by adding a few drops of sulphuric acid at the end of the operation. After filtration, a slight excess of precipitated calcium carbonate, followed by potassium chromate, is added to the liquid, and titration is performed by decinormal nitrate of silver. The method is found exact to within 0.2 per cent. in the case of 48 per cent. of potash. C. S.

Cuprous oxide as a Reagent for Nitrites. P. Sabatier. (*Comptes rendus*, cxxii. [24], 1417.)—The addition of a little red cuprous oxide to a dilute solution of sodium nitrite in concentrated sulphuric acid produces a fugitive, but intense, violet purple coloration, consisting of the cupric salt of nitrodisulphonic acid, and the same effect is produced (more gradually) by all the cuprous or cupro-cupric compounds, but not by cupric salts. This reaction is employed by the author as a test for nitrous acid, a drop of sulphuric acid being added to a drop of the suspected liquid, a few grains of cuprous oxide being then added to the mixture; it is less sensitive than Griess' reagent, and is hindered by the presence of nitric acid in quantity.

C. S.

Natural and Industrial Phosphates—III: The Insoluble Residue. H. Lasne. (*Ann. de Chim. Analyt.*, i. [11], 207.)—The insoluble residue from the hydrochloric solution (ANALYST, xxi., p. 195) is calcined on a tared filter, the loss in weight indicating organic matter insoluble in acid. The calcined mass is usually white, containing only minute traces of iron, and is generally composed of granular and amorphous silica, with silicates of potassium, sodium and alumina. When fluorine is present in the phosphate, allowance must be made—at the rate of 0.3846 of silica to 1 of fluorine—for the silica volatilized as fluoride, provided there is an excess of silica present. In other cases a weighed amount of silica (some 5 per cent.) should be added to the phosphate at the outset and deducted from the final results. The residue, being generally free from lime and iron, may be treated by the usual methods for dissociating silicates, the simplest being to drive off the silica by hydrofluoric acid, and take the loss in weight after the addition of a few drops of sulphuric acid and re-calcination. The new residue is dissolved in sulphuric acid and tested for alumina, alkalis, phosphoric acid (from insoluble amblygonite, monagite, etc.), titanate acid, and zirconium—the presence of rare elements affording assistance in determining the origin of the phosphate. Barium should also be looked for, its presence indicating adulteration of the (bone) superphosphate by plaster of Paris, which substance, according to the author's statement, contains a considerable quantity of barium sulphate.

C. S.

Volumetric determination of Mercury by Potassium Iodide. G. Denigès. (*Bull. Soc. de Pharm. de Bordeaux*, April, 1896, p. 97; through *Ann. de Chim. Analyt.*, i. [11], 211.)—This method is based on the double decomposition of mercuric salts and potassium iodide. A deci-normal solution of mercuric chloride is prepared by moistening 13.55 grammes with 5 c.c. of hydrochloric acid, agitating with 10 c.c. of water and making the volume up to 1 litre. The iodide solution is made by dissolving rather more of the salt than is theoretically necessary, and titrating it with the mercuric solution in presence of a couple of drops of sodium bisulphite (36-40° B_é). The addition of 1 c.c. of ammonia increases the stability of the reagent. Taking a as representing the number of c.c. of deci-normal mercuric solution required to titrate 10 c.c. of the iodide solution, 100 ($a - 10$) c.c. of water must be added to a litre of the iodine solution, in order that it may correspond to the $\frac{1}{10}$ -normal solution of mercury. The corrections for dilutions of the reagent between $\frac{1}{10}$ and $\frac{1}{100}$ -normal strength are calculated as follows, a having the value already stated:

between $\frac{1}{10}$ and $\frac{1}{100}$ -normal	$x = \frac{(a-10)^2}{1,000} \times 4.$
" $\frac{1}{10}$ " $\frac{1}{100}$ "	$x = \frac{a^2}{1,000}$
" $\frac{1}{100}$ " $\frac{1}{1000}$ "	$x = \frac{a-25}{10}$

The results will correspond very closely to the theoretical quantities, provided the liquid is neutral or contains acid not exceeding 0.05 c.c. of HCl.

The mercury is brought to the condition of chloride by the action of nitrohydrochloric acid, there being practically no loss of mercury by evaporation, if the liquid is strongly acid and the operation is performed in a tube.

To apply the test to a mercurial solution, 1 gramme of substance is placed in a test-tube with 2 c.c. of hydrochloric and 1 c.c. of nitric acid (double these quantities in the case of vermilion) and boiled until it has dissolved and a cloudy precipitate commences to form, whereupon 5 or 6 c.c. of water are added, and the whole made up to 100 c.c. The solution is titrated by noting the volume required to impart a feeble red coloration to 10 c.c. of the iodine solution containing two drops of bisulphite; if more than 14 c.c. are used, a correction, on the lines given above, must be made. By dividing the amount of mercuric solution into 1,000, the percentage titre of mercury in the substance will be obtained.

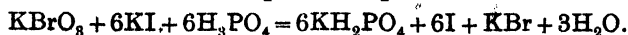
The results cited approximate very closely to the truth, the iodides of mercury, however, giving figures somewhat low on account of the formation of iodic acid, which is reduced by the bisulphite to an iodide of the alkali base, and falsifies the result of the titration.

C. S.

Decomposition of Alcoholic Indicator Solutions. Verbiessé. (*J. fabr. sucre*, 1896, xxxvii., 20; through *Chem. Zeit. Rep.*, 1896, 171.)—The author remarks that acid solutions of phenolphthalein and other indicators in alcohol often become useless in a few days, and lead to erroneous results owing to the formation of acetic acid.

F. H. L.

Volumetric Process for the Determination of Phosphoric and Arsenic Acids. A. Christensen. (*Nordisk pharm. Tidsskrift*, 1896, iii., 77; through *Chem. Zeit. Rep.*, 1896, 153 and 171.)—This process depends on the reaction:



For the practical analysis of phosphates 4 to 5 grammes of the sample are dissolved in 20 to 30 c.c. of 1·2 nitric acid, and diluted to 500 c.c. Twenty to fifty c.c. are treated with 1 to 2 grammes of silver nitrate, either solid or as a concentrated solution, and normal soda is dropped in until the yellow precipitate no longer disappears. Ten per cent. ammonia is then added till the liquid is alkaline, and the whole is boiled for a few minutes and allowed to settle. The precipitate is filtered off, washed free from lime (using at the last a 2 or 3 per cent. solution of potassium nitrate), and returned to the original flask by the aid of the same liquid. A solution of 0·3 to 0·6 gramme of common salt is introduced, and the mixture warmed slightly. The solution of the sodium phosphate is filtered into a stoppered flask, the residue washed till neutral with the nitrate liquid, 10 c.c. of seminormal sulphuric acid, 3 grammes of potassium iodide, and 10 c.c. of 5 per cent. potassium bromate added, and the whole made up to 100 or, at most, 120 c.c. After standing for half an hour at 40° or 50° C., it is ready for titration. In the analysis of basic slags the magnesium precipitate is washed first with 2·5 per cent. ammonia, then with 90 per cent. alcohol, rinsed into a stoppered flask, and 30 c.c. of decinormal sulphuric acid added for every 0·1 gramme of phosphoric anhydride it contains; the liberated acid being estimated as above.

In the case of arsenic acid, good results may be obtained by precipitation with magnesia mixture, Puller's correction for the solubility of the double salt in weak ammonia being employed.

The volume of the liquid before titration must always be kept uniform.

F. H. L.

APPARATUS.

Bunge's Improved Analytical Balance. (*Chem. Zeit.*, 1896, xx., 326.)—In the ordinary process of weighing much time is wasted, and considerable harm is done to the agate bearings by the oscillations of the beam during the earlier attempts at attaining a balance. This improvement consists in the addition of a separate arrangement under the left-hand (object) pan, by means of which the weight of the substance is read directly to the nearest gramme on a scale attached to any convenient spot in the case. This pan is carried by a gilt steel stirrup made so as to expand in length according to the weight of the object. Underneath it is an agate point fastened to the arresting gear of the pan, which depresses to a greater or less extent an adjustable agate cup fixed to the shorter end of the lever-balance, the opposite extremity of which carries the pointer travelling over the scale. In the case of balances that are always swinging, enough space is left to permit of this movement; but in the other variety the addition is thrown in and out of gear by an eccentric. The improvement is patented in Germany.

F. H. L.

Araeometer Pipette. Greiner and Friederichs. (*Zeit. anal. Chem.* 1896, xxxv., 169.)—This consists of a peculiarly shaped pipette in the body of which is inserted a small hydrometer of toughened glass. It is especially suitable for the rapid determination of the specific gravity of liquids, of which only a small quantity is available. Owing to the small size of the hydrometer the scale is necessarily limited.

C. A. M.

Standardizing Volumetric Apparatus. L. L. de Koninck. (*Chem. Zeit.*, xx., 1896, 460.)—The author draws attention to the inexact way in which measuring cylinders are usually graduated, and insists on each piece of apparatus being tested before use. He observes that burettes, instead of being marked in true cubic centimetres, are often divided into what he calls millimohrs—the apparent volume of 1 gramme of water at 15° C., as weighed in the air with brass weights (100 millimohrs = 100.2 c.c.). For a quick test as to whether due care has been taken in the graduation, he suggests that the distance between the 0 and the 10 mark should be measured with a pair of compasses. If the other corresponding intervals are the same, the tube may at once be rejected, for it is impossible to manufacture glass tube with strictly parallel sides.

For the calibration of tubes closed at one extremity, the process of inverting them and allowing water to run in from a standardized burette is useless, since the meniscus, being inverted, causes the volume of the liquid introduced to appear quite different from its true amount. To perform the operation correctly, the cylinder should be provided with a rubber cork having two holes. Through one is inserted a tube of 1 m.m. internal diameter drawn out to a fine point inside the vessel, bent upward again to a U-shape on the outside, and closed with a rubber tube and screw-clamp. The second hole carries a straight tube of the same size, drawn out to a point after the manner of the jet of a Mohr's burette. The vessel is completely filled with water, and the stopper carefully inserted so as not to introduce any air. It is next fixed in the normal position, and the clamp opened until the former tube is exactly filled with air. Successive volumes of liquid may then be run off either into a tared beaker and weighed, or simply into a correctly graduated burette. The process can be modified in a variety of ways, and if proper care is exercised in keeping the temperature normal, it will be found very exact.

F. H. L.

REVIEW.

"PRINCIPLES AND PRACTICE OF AGRICULTURAL ANALYSIS." By HARVEY W. WILEY.
Volume I.: Soils. Volume II.: Fertilisers. Chemical Publishing Company, Easton, Pa.

IN March, 1894, we had the pleasure of noticing the first monthly number of a new work by Prof. Wiley, bearing the above title. The treatise, as might have been expected, has outgrown the proportions anticipatorily assigned to it by the author, and is not yet complete. We have before us, however, the two volumes already

completed, the first dealing with soils and the second with fertilisers. The chemistry and analysis of farm products and feeding stuffs is deferred till the third volume, still in course of publication.

The two volumes already published do not disappoint the promise given by the work in its infancy. The volume on soils is a storehouse of valuable information, not the least acceptable part of which is to be found in the liberal bibliographical references appended to each part, each reference being connected by a key number with the portion of the text to which it relates.

As an instance of the conscientious spirit in which the book is written, it may be mentioned that no fewer than twenty pages are devoted to the various methods of collecting samples of soil, and to the various considerations affecting this important initial step towards soil analysis. Such frequently neglected subjects as soil thermometry are also treated. Methods for investigating the cohesive and adhesive properties of soils, their porosity and absorptive powers, and their capillary properties as regulating water movement, as well as their permeability, both as regards water and air, are discussed at length. So also are modes of investigating the selective absorptive power of soils for potash and other chemical constituents of manures.

The subject of mechanical analysis also occupies a great deal of the author's attention, and we have here, for the first time, brought together a summary of modern improvements upon the old crude methods of mechanical analysis directed towards obtaining results possessing the advantage of strict comparability as well as of practical meaning. A very beautiful set of micro-photographs of sand of various qualities and degrees of coarseness, silt, clayey particles, etc., appropriately illustrates this part. The gases in soils also come in for much detailed attention.

Of the nearly six hundred pages comprising the volume on soils, the first three hundred are almost entirely devoted to such physical aspects of soil examination as have been referred to, the remaining half being devoted to chemical analysis. The portion on the chemical analysis of soils deals not only with the determination of the total percentages of the various constituents, but also with the methods devised by various investigators for the attempted estimation of the "available" constituents, such as the quantities soluble in water, carbonic acid water, weak ammonia salts, acetic acid, weak citric acid, etc. The determination of the nitrifying power of soils, as based upon bacteriological culture methods, and the investigation of the distribution of the nitrifying organisms in the various layers of the soil, are discussed at some length in a special part, along with the origin and estimation of oxidised nitrogen in soils, and in rain and drainage waters.

Any agricultural chemist who can read through the volume on soils without adding to his stock of knowledge, must be enviably well versed in his subject.

In the volume on the analyses of fertilisers, we have brought together, in a compact space, a large number of processes which have obtained more or less professional recognition. The actual practitioner in agricultural analysis will be glad to see collated a considerable number of the arbitrary "ammonium citrate" methods for the determination of reverted and available phosphates in various countries, as laid down by custom or by official convention. Prof. Wiley proposes that the words "available" and "reverted" should be abolished, as sometimes begging an unsettled

question, and suggests the use of the words "citrate soluble" to describe the phosphates dissolved in such processes. It is only necessary, however, to glance through the records which he has brought together of experiments with such methods, to realize how variable are the results yielded, depending as they do, not merely upon the acidity or alkalinity of a difficultly neutralizable reagent, but also upon the actual quantity of reagent used, and upon the time and temperature of digestion. He very properly points out that comparative results are only to be obtained by rigid adherence to the details of the particular process of which circumstances may dictate the choice.

Prof. Wiley appears to omit reference to what some chemists venture to consider a much more satisfactory reagent than ammonium citrate, viz., a weak (1 per cent.) solution of citric acid, as suggested by Tollens and Stutzer a dozen years ago. More recent investigation has shown this solution to have approximately the acidity of the root sap of plants. Its application to soils is, as has already been mentioned, dealt with in the volume on soils, but its applicability to the analysis of fertilisers has either been overlooked by the author, or—as seems more probable—he considers that the ammonium citrate process, modified in some way or other, is too firmly established for displacement by the more simple and apparently more rational citric acid method.

In a short section on the manufacture of superphosphate, it is satisfactory to see that the author does not repeat the old text-book error, still fashionable, of stating that the "soluble phosphate" in superphosphate consists merely of tetra-hydric monocalcic phosphate, but points out that, as first shown by Ruffe, when excess of acid is used, as in ordinary manufacture, free phosphoric acid constitutes at least a considerable part of the soluble phosphatic matter.

The matter on nitrogen determination is necessarily and properly voluminous, and includes descriptions of the various modifications of Kjeldahl's process.

Perhaps the least satisfactory feature of the book is the section on the determination of iron and aluminium oxides in raw phosphates. A number of methods are given, all of which, excepting that of Hess, direct that the raw material shall be dissolved in nitric acid, with or without the addition of hydrochloric acid. This serious error, which is involved in the Glaser as well as other processes, results in the inclusion of iron pyrites with oxide of iron, a substance to which it is not technologically analogous, inasmuch as pyrites is scarcely, if at all, attacked by the acids used in superphosphate making, whereas iron oxide is so attacked, and vitiates the keeping power of the superphosphate. This was fully pointed out by Shepherd in *THE ANALYST* for November, 1893, and English analysts cannot fail to regret that Prof. Wiley has not drawn attention to this very important matter, particularly as neglect of this consideration has led to serious under-valuation, more particularly of certain descriptions of American phosphates, which contain considerable quantities of perfectly harmless pyrites. As such phosphates form a subject of international trade between England and America, it seems a pity that, in a work which will take so highly authoritative a place as that by Prof. Wiley, this source of error should not have been pointed out, with a view to inducing American analysts to assimilate their practice to that of English chemists by using simple hydrochloric acid as a solvent, and thus to differentiate between pyrites and iron oxide.

It is also to be regretted that the only modification of the acetate method for

oxide of iron and alumina, is the somewhat cumbrous and not very satisfactory one of Hess. The acetate method has been largely discarded, not on account of any inherent untrustworthiness, but on account of the loose and unwarranted assumption, so long made with regard to it by many chemists, that the ammonium acetate precipitate consisted merely of ortho-phosphates of iron and aluminium. Experienced employers of the process, who have not fallen into the error of ignoring the presence of lime in the ammonium acetate precipitate, and who, after weighing every precipitate, have taken the precaution to actually determine the lime and phosphoric acid in it, have never had to complain of its untrustworthiness, though no doubt the accurate analysis of the precipitate necessary for correctly arriving at the results involves somewhat nicer manipulation, and is somewhat more tedious than the Glaser process. It almost invariably gives results identical with the Glaser process—provided, of course, that the Glaser process is modified by the substitution of hydrochloric for nitric acid or nitro-hydrochloric.

It may further be remarked that it appears to be not satisfactory, in these days of accurate commercial analyses, that chemists should allow themselves (except for rough and ready work in the factory) to assume that half the weight of the mixed phosphates of iron and aluminium consists of the bases in question. Aluminium phosphate contains 42 per cent. of aluminium oxide, while the percentage of ferric oxide in ferric phosphate is nearly 53. In commercial contracts, the permissible limits of iron and aluminium oxides in phosphates are rigorously laid down, and even a few tenths per cent. materially affect the invoice value of the cargo of phosphate. The assumption that exactly half of the mixed "Glaser" phosphates consists of iron and aluminium oxides—altogether irrespectively of the proportions in which these two bases are present—may well be sufficient to lead to consequences financially serious to either buyer or seller, and even to the rejection of unfair cargoes. The right of rejection often lying with the buyer if a given limit, such as 4 per cent. of the mixed oxides, is overstepped. It does not involve much time or trouble to determine the phosphoric acid in the Glaser precipitate, and thus obtain correctly the real quantity of the oxides with which it is combined.

It is, however, not given, even to so careful and experienced an author as Prof. Wiley, to include in one treatise all that may be desired on every subject of which he treats, and attention is drawn to these points in the hope of seeing them dealt with in a future edition, and not in any spirit of inappreciation of a work which can scarcely fail to take an indispensable place amongst the standard books of reference in the library of the specialist. As a practical guide-book, it is perhaps scarcely a book for the beginner; for while the author details processes, and states when and where they have been officially recognised, he leaves the operator to make his own selection, somewhat after the manner of Fresenius. To presuppose that his choice will be wise, is to presuppose the existence of experience and the faculty of acquired practical intuition that experience alone develops.

Analysts, both in England and in America, owe Prof. Wiley their gratitude for these two volumes, and will look forward with pleasurable anticipation to the completion of the third volume.

B. D.

THE ANALYST.

OCTOBER, 1896.

WHITE-WINE VINEGAR.*

By ALFRED H. ALLEN.

WHITE-WINE VINEGAR was originally understood to mean a vinegar made from wine derived from white grapes. Of late years the sale of the genuine article has greatly declined, and distilled malt vinegar or simple diluted acetic acid has been very commonly sold in its place. This practice, though very general, has not the sanction of universal custom, since some of the leading dealers always supply real white-wine vinegar when asked for it.

The following are analyses of two samples of the real article purchased retail in a town in the Midland counties :

<i>Parts per 100 measures :</i>						A.	B.
Acetic acid	6.37	6.49
Extractive matters	1.42	1.55
Containing mineral matters	0.28	0.30
With alkali (K_2O)	0.046	0.046

Two other samples obtained direct from the importers, and which I believe to be specimens of genuine white-wine vinegar, showed on analysis :

Specific gravity						C.	D.
...						1.0197	1.0211
<i>Parts by weight per 100 measures :</i>						A.	B.
Extractive matters.	Acetic acid	7.98	7.78
	Albuminous matters	0.10	0.19
	Carbohydrates, etc.	1.65	1.65
	Mineral matters	0.20	0.53
Containing phosphoric acid						trace	0.065

Genuine wine vinegar always contains a notable quantity of acid potassium tartrate, which is not present in vinegar from other sources.

The following samples were procured by retail purchase from pharmacists in Midland towns, "half a pint of white-wine vinegar" being asked for in each case. The results showed that dilute acetic acid or distilled malt vinegar, sometimes coloured a pale yellow, was supplied in each case. The amount of extractive matter was insignificant. The price of the samples varied from 1½d. to 4d. per half-pint.

* Abstract of a paper read before the British Pharmaceutical Conference.

Number.	Label on Bottle.	Acetic Acid. Grammes per 100 c.c.	Colour.
1	Pure white-wine vinegar ...	6.45	Water-white
2	Vinegar	4.41	Pale straw
3	White vinegar	5.01	Nearly colourless
4	Acetic acid vinegar ...	3.15	Water-white
5	Vinegar	4.80	Water-white
6	White-wine vinegar ...	5.47	Nearly colourless
7	Distilled malt vinegar ...	5.22	Water-white
8	White-wine vinegar ...	5.10	Water-white
9	White-wine vinegar ...	5.10	Water-white
10	Distilled vinegar ...	5.91	Water-white
11	White-wine vinegar ...	4.23	Pale straw
12	No label	5.24	Water-white
13	White-wine vinegar ...	5.79	Water-white
14	White-wine vinegar ...	6.93	Water-white

White-wine vinegar has the colour of sherry. It has a vinous aroma and peculiar flavour. However much it may be appreciated as a condiment by those who obtain the real article, it is clearly waste to employ it for pickling purposes, since its distinctive characters are thereby lost.

It seems highly improbable that wine vinegar will ever again come into general use in this country, and it probably would completely disappear if distilled acetic acid were not supplied in its place, or if the label described correctly the nature of the article sold. Where distilled vinegar is substituted, according to the supposed requirement of the purchaser, it might with advantage be labelled, "Distilled vinegar; commonly called white-wine vinegar."

NOTE ON THE MICROSCOPIC DETECTION OF BEEF FAT IN LARD.

By THOMAS S. GLADDING.

In the preparation of crystals of lard and beef stearin for microscopic examination I find the following method gives excellent results, the crystals being of good size and of distinctive form. Dissolve 5 c.c. of melted lard in a mixture of 20 c.c. absolute alcohol and 10 c.c. ether in a small Erlenmeyer flask, heating gently if necessary. Place a plug of cotton in the mouth of the flask, and allow to stand in a cool place for about half an hour. The stearin crystallizes out, the olein remaining in solution. Filter rapidly through a paper wet with alcohol, using a filter-pump, and wash crystals and paper once with the above alcohol-ether mixture (10 : 5). Let the crystals dry in the air, and remove them from the paper to the flask. Dissolve in 25 c.c. of ether, replace the cotton plug, and place the flask in a slanting position in a large beaker (about 1 litre) nearly full of water. Keep this in a cool place over night. The ether evaporates very slowly, and the crystals of stearin are gradually formed in the solution, the large quantity of water surrounding the ether solution guarding against any sudden change of temperature. For valuable plates giving characteristic forms of lard stearin crystals and beef stearin crystals reference is made to Bulletin No. 13, Part IV., Division of Chemistry, United States Department of Agriculture.

NOTE ON AMMUNITION BREAD.

By SIR CHARLES A. CAMERON, PROFESSOR OF CHEMISTRY, R.C.S.I., MEMBER OF THE ARMY SANITARY COMMITTEE.

THE amount of water in bread varies from 38 to 45 per cent., and is generally about 40 per cent. I have met with two specimens lately which contained larger proportions of water than I had hitherto met with or read of. The first was a loaf—part of the bread supplied by contract to the troops stationed in Cork in June last. It contained per 100 parts :

Water	58.29
Organic matter	40.26
Ash :						
Soluble	0.45
Insoluble	1.00

100.00

This bread was extremely sour, contained lumps of half-baked dough, and was full of cavities. The large percentage of insoluble ash showed that the flour used was of the lowest class.

A specimen of the bread supplied in August, 1896, to the troops at Clonmel, County of Tipperary, resembled the foregoing. It contained per 100 parts :

Water	58.28
Organic matter	40.57
Ash	1.15

100.00

This bread was very sour, like a sponge, and very dark in colour.

I have never met with such inferior bread before ; and if such were supplied to troops on the march they would not be able to travel far upon it. It is an old saying that soldiers march on their stomachs.

In a specimen of bread just received from the military authorities of Clonmel the proportion of water was only 39 per cent. Some ammunition bread from Cork was also greatly improved as regards proportion of water and other respects.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD ANALYSIS.

The Microscopy of Honey. K. Dieterich. (*Helpfenberger Annalen*, 1895-96, 47 ; through *Chem. Zeit. Rep.*, 1896, 156.)—In spite of the statements that artificial honey can be distinguished from the genuine product by the absence of pollen grains, the author finds that ordinary forms are present, and in addition to these, a special and characteristic form never met with in Dalmatian or American honey. This particular variety is in large excess of the normal forms, and fills the whole field of

view, only isolated specimens of the others being observable. Artificial honey exhibits the same crystals of cane-sugar and glucose as the genuine substance, but the cane-sugar crystals are very few in number.

F. H. L.

Estimation of Sugar in Chocolate. X. Rocques. (*Ann. de Chimie Analyt.*, i. [15], 288.)—In order that the proportions of ash, fatty matter, etc., found in chocolate may be referred to the original cacao, it is necessary to estimate the added sugar. This may be done by mixing 15 grammes of the grated chocolate in a flask with 90 c.c. of distilled water, raising the temperature to 40° C. to melt the fat and produce an emulsion. After agitating for a moment, add 15 c.c. of a 10 per cent. solution of subacetate of lead and filter, collecting 70 c.c. (= 10 grammes of substance), and throwing down the lead therein by a mixture of 20 c.c. of a 20 per cent. solution of sodium sulphate and 10 c.c. of glacial acetic acid (unless the reagents be added together the precipitation is incomplete), leaving the glucose and sugar in solution. The glucose is estimated direct from a few c.c., and the saccharose by heating 50 c.c. for three hours on the water-bath, and treating with Fehling solution after making up the volume to 500 c.c. The acid in the liquid suffices to invert the saccharose, and, although requiring longer than inversion by hydrochloric acid, has the advantage of not acting on any dextrin that may have been added as an adulterant. By subtracting the weight of glucose from the total sugar, and multiplying the remainder by 0.95, the amount of crystallizable (added) sugar is obtained, which should average 55 per cent. of the chocolate. Ratios of 60 per cent. and thereabouts indicate additions of sugar to conceal inferiority of the cacao used and reduce the selling-price. C. S.

On the Quantitative Estimation of Cellulose in Foods. G. Baumert. (*Zeit. angew. Chem.*, 1896, 408-411.)—The chief novelty in the author's process is the filtration of the cellulose through asbestos previously washed with acid and alkali, and arranged in a thin layer on a filter disc in a funnel. Two grammes of the finely-divided, air-dried substance (*e.g.*, meal) are placed in a beaker of about 200 c.c. capacity, moistened with 90 per cent. alcohol, and, after the addition of some dry, pure asbestos, mixed with 100 c.c. of dilute sulphuric acid (12.5 grammes per litre). The mass is well stirred, covered with a glass, and placed in a water-bath, so that the beaker is only surrounded by steam. After an hour, with occasional stirring, the mass is filtered through asbestos, the filter washed with hot water, and the whole of the asbestos, with the residue, returned to the beaker.

The contents of the beaker are next digested with dilute soda solution (12.5 grammes per litre) for an hour in the water-bath, and filtered through a new asbestos filter, which is then washed successively with hot water, alcohol, and ether. The asbestos is transferred to a platinum basin, dried at 100° C., and weighed. It is then ignited until all carbon has disappeared, and again weighed, the difference between the two weighings giving the amount of cellulose. When the material to be examined is very rich in fat, as in the case of cocoa, a preliminary extraction, with 30 c.c. of 96 per cent. alcohol, is made by placing the beaker containing the 2 grammes of

substance in the water-bath. The alcohol is filtered through asbestos, the extraction and filtration repeated once or twice with more alcohol, the asbestos filter and its contents placed in the beaker, and the extraction, with sulphuric acid and soda, carried out as before.

The following table gives some of the results obtained in this way:

Per Cent. Cellulose.			Per Cent. Cellulose.		
Wheat meal, Russian ...	0.00	0.10	Rye bran ...	4.90	5.05
„ „ La Plata ...	0.25	0.30	Wheat bran ...	7.20	7.30
„ „ fine groats ...	0.15	0.20	Wheat bread ...	1.32	1.42
„ „ coarse „ ...	0.20	0.20	Malt coffee ...	10.60	10.80
Rye meal, No. O. ...	0.15	0.20	Chicory coffee ...	6.65	6.76
„ „ No. II. ...	1.15	1.15	Coffee, roasted ...	12.45	12.45
„ „ Fodder ...	1.80	2.00	Cocoa husks ...	16.30	16.35
			Potatoes ...	0.44	0.46

If the time of digestion be lengthened the percentage of cellulose naturally falls; but the author considers the results obtained by digesting for one hour as the most correct.

C. A. M.

Examination of Peppers. F. Bauer and A. Hilger. (*Forschungsber. Lebensmittel*, etc., 1896, iii., 113; through *Chem. Zeit. Rep.*, 1896, 156.)—The best method for testing the purity of pepper depends on the estimation of the piperine, either in the form of piperic acid or by titration as piperidine. Genuine peppers contain between 5.55 and 7.77 per cent. of the alkaloid, while in the husks it only amounts to 0.2 per cent.; therefore any sample containing less than 4 per cent. may be considered adulterated. The author has also found that the amount of furfuralhydrazone yielded by 5 grammes of dry black pepper is 0.20 to 0.23 gramme, in white pepper 0.046 to 0.052, and in dust and husks 0.41 to 0.56 gramme, so that an addition of substances of a similar nature more than 15 per cent. in extent can be detected. The samples must always be examined microscopically also. “Long pepper” contains 4.9 per cent. of piperine, and yields 0.185 to 0.197 gramme of furfuralhydrazone. Its ash is 6.02 to 6.80 per cent. in weight.

F. H. L.

ORGANIC ANALYSIS.

The Electrical Conductivity of Fats and Oils. L. Herlant. (*Bull. de l'Ass. belge*, 1896, x., 48-54.)—Salt solutions offer different resistance to an electric current, the amount of which is constant under the same conditions for one and the same substance. The *specific resistance* of a solution is the measurement for a cube with a side of 1 cm., whilst the inverse function of this $\frac{1}{r}$ gives the *specific conductivity*. To simplify the process and to determine the *electrolytic capacity of the trough*, a typical salt solution of known conductivity is employed, and the subsequent results based on this. For this purpose the author used a $\frac{1}{500}$ N solution of potassium chloride

(1.49 gramme per litre), the conductivity of which salt was determined by Kohlrausch at 0.002244 for a cubic centimetre at 18° C.

In the formula $\frac{1}{r} \times k = 0.002244$, k represents the capacity of the trough, and when the potassium chloride solution is replaced by the solution to be examined, the distance between the electrodes is modified, which gives the specific conductivity l of the solution.

$$l = \frac{1}{r} \times k.$$

For the examination of fats and oils, 10 grammes, which must be dry and free from salts, casein, etc., are mixed in a flask with 45 c.c. of pure normal alcoholic potash, and saponified by heating for thirty minutes on the water-bath under a reflux condenser. When saponification is complete, the soap solution is made up to 250 c.c. with distilled water and electrolysed.

The following table gives the results obtained in this way by the author :

Substance.	Butyro-refractometer. Zeiss at 35° C.	Reichert-Meissel No.	Critical Temperature of dissolution.	Specific Conductivity at 18° C.
Butter 1	... 44.5	28.7	98° to 103°	0.006457
„ 2	... 45	28		0.00650
„ 3	... 45	28.6		0.006507
„ 4	... 48	25		0.00701
Margarine 1	... 52.5	3.2	122° to 123°	0.008221
„ 2	... 56.5	1.9		0.008459
„ 3	... 54	2		0.008472
„ 4	... 58	—		0.008489
Cotton oil	... 63	—	115° to 116°	0.008629
Earthnut oil 1	... 58.25	—		0.00870
„ „ 2	... 60.25	—		0.008741
Sesame oil	... 63	—		0.008779
Olive oil 57.25	—	123°	0.009927

These figures show that the conductivity stands in direct relation to the refractive index and critical temperature. After being left six weeks, soap solutions made from butter and margarine showed a slight decrease in conductivity. The author believes that with more data the method will prove of use in detecting adulteration in butter, though in the case of oils the results are not so promising.

C. A. M.

A Colour Reaction for Earthnut Oil. A. Van Engelen. (*Bull. de l'Ass. belge*, 1896, x., 161-162.)—The reagent is a solution of 0.25 gramme of sodium molybdate in 20 c.c. of concentrated sulphuric acid, and is called the molybdic reagent. Or Fröhde's reagent recently prepared may be used. To about 5 c.c. of the oil, in a test tube, five or six drops of the molybdic reagent are added and the tube shaken. Earthnut-oil gives a violet-purple colour, fading after about a minute. With Fröhde's reagent the reaction is somewhat different. With the following oils the results were :

	Molybdic Reagent.		Fröhde's Reagent.	
	Without Shaking.	With Shaking.	Without Shaking.	With Shaking.
Olive ...	Brown	Mahogany brown	Brown	Mahogany brown
Sesame...	Dark brown	Dark brown	Dark brown	Dark brown
Cotton ...	Mahogany brown	Black	Black	Black
Poppy ...	Greenish yellow	Lilac	Yellow	Yellow with violet tint
Earthnut	Greenish yellow	Purple	Greenish	Violet

A mixture of olive and earthnut oils did not give a satisfactory result.

C. A. M.

The Leffmann-Beam Process for the Estimation of Volatile Fatty Acids.

W. Karsch. (*Chem. Zeit.*, 1896, xx., 607.)—This process was introduced by Dr. Vieth into the "Milchwirtschaftliche Institut" at Hameln two years ago, and has been in constant use ever since. It is not only much quicker than the method of alcoholic saponification, but, as the figures in the annexed table show, it gives more uniform results, which, though always lower than those obtained by Wollny's process, are evidently the more correct.

It is carried out as follows: One hundred grammes of caustic soda are dissolved in an equal weight of water, and 20 c.c. of the solution mixed with 180 c.c. of pure concentrated glycerin. Five grammes of the filtered fat are weighed into a 300 c.c. Erlenmeyer flask, 20 c.c. of the alkali added, and the vessel held by rubber-covered tongs over a naked flame for three or four minutes, until the water is driven off and the liquid becomes clear. It is next cautiously diluted with 135 c.c. of water free from carbon dioxide, and when the soap has dissolved, a fragment of pumice and 5 c.c. of 1:4 sulphuric acid are dropped in. The liquid is then ready for immediate distillation.

The following samples of butter were saponified in this way, and also with alcoholic alkali according to Wollny's directions; but, owing to the difficulty of removing the last traces of alcohol in the time given (thirty to forty-five minutes), Samples 1 to 7 were allowed to remain for five minutes on the water-bath with the flasks open, while 8 and 9 were distilled without opening for one and a half to two hours. No. 10 was also tested by Sendtner's process, and gave 28.85. Blank analyses showed the necessity for corrections corresponding to 0.2 to 0.3 c.c. of the decinormal soda; these have in all cases been made.

Sample No.	Glycerin Process.		Alcoholic Process.	
1.	28.18	28.29	29.18	29.39
2.	27.96	28.07	28.63	29.07
3.	27.96	28.07	28.86	28.96
4.	27.08	27.08	27.97	28.08
5.	28.18	28.29	29.29	29.51
6.	28.12	28.18	28.41	29.18
7.	27.74	27.74	28.08	28.22
8.	27.79	27.85	27.86	28.02
9.	28.18	28.18	28.30	28.68
10.	28.00	28.07	28.41	28.68

To investigate the influence of carbon dioxide, another solution containing the soap from Sample 10 was blown into three times, which caused the Reichert-Meissl number to rise from the values recorded to 29.17. The amount of water used for solution of the soap must also be adhered to, for experiments on Sample 9, when only 115 c.c. were employed, gave a mean result of 29.28.

The author remarks that of all the samples of butter he has examined by the glycerin process since last October, the highest Reichert-Meissl number has been 30.82, and the lowest 26.53.

F. H. L.

The Analysis of Wool-Fat. F. Ulzer and H. Seidel. (*Zeit. angew. Chem.*, 1896, 349-350.)—Not being able to obtain completely concordant results in the saponification of wool-fat, even when carried out under pressure, the authors propose to determine the "total acidity number" instead of the saponification number, as was recommended by Benedikt and Mangold in the case of beeswax. The "total acidity number" (Benedikt, *Analyse der Fette*, p. 441) is the amount of potash in tenths of a per cent. required to neutralize the mixture of fatty acids and fatty alcohols obtained by saponifying the wax (or wool-fat) and decomposing the soap with hydrochloric acid. To determine it, 20 grammes of potash are dissolved in 20 c.c. of water in a porcelain basin holding from 350 to 500 c.c., and the solution heated to boiling. Twenty grammes of the melted wool-fat are then stirred in, and the whole heated to boiling for about a minute, the heating continued on a water-bath until a thick uniform soap is obtained, and the basin finally placed for two hours in the water-oven to complete the saponification. The soap is dissolved in about 250 c.c. of boiling water, and decomposed with 40 c.c. of hydrochloric acid previously diluted with water. The clear fatty layer is repeatedly washed with boiling water until the washings are free from acid, and then dried in the water-oven. From 5 to 6 grammes of the dry mixture of fatty acids and alcohols are warmed with acid-free alcohol on the water-bath and titrated with standard alkali, phenolphthalein being used as indicator.

The "total acidity number" of a sample of Australian wool-fat determined by the authors was (1) 100.2, (2) 100.9, and (3) 101.9, whilst the results obtained with a South American wool-fat were 96.4, 96.7, and 96.9. The Reichert-Meissl numbers varied between 6.7 and 9.9.

The authors conclude that for the technical analysis of wool-fat sufficient data are furnished by the determination of the acid value, the "total acidity number," the iodine number, and the Reichert-Meissl number. A gravimetric estimation of the unsaponifiable matter is also advisable.

C. A. M.

Estimation of Resin in Fatty Oils. P. Cornette. (*Ann. Pharm.*, 1896, ii., 240; through *Chem. Zeit. Rep.*, 1896, 192.)—This process depends on the fact that the sodium salts of the higher fatty acids are insoluble in a saturated solution of common salt, while the corresponding resinates are perfectly soluble. Ten grammes of the oil are saponified, the soaps dissolved in warm water, allowed to cool, and an

excess of a strong solution of sodium chloride added. The mixture is filtered cold, and the precipitate washed with salt solution. The filtrate, which should be clear, is acidified with sulphuric acid, and the resin acids collected and weighed. In the case of pure linseed-oil a slight turbidity is often observed, but it cannot be mistaken for the precipitate produced by resin acids.

F. H. L.

On Resinated Metallic Oxides. H. Amsel. (*Zeit. angew. Chem.*, 1896, 429-433.)—These preparations, which now play such an important part in the manufacture of varnish, are made either by melting the resin and the oxides of lead or manganese together, or by saponifying the resin with potash, and precipitating the resinate from the aqueous soap solution by means of a lead or manganese salt.

Colophony, which on account of its cheapness is the principal resin employed, is prepared from pine-tree resin by removing the turpentine-oil and water by heat, and then keeping the residue in a melted state until it becomes perfectly clear. According to Wiesner, this clarification is due to the change of crystalline abietic acid into its amorphous anhydride. The best varieties are quite free from crystals; in poorer qualities bow-shaped crystals can be observed under the microscope. The colour varies from yellow to dark-brown, according to the time of heating and degree of heat. In several concordant determinations with three differently coloured kinds of colophony the author obtained the following values:

	Acid Value.	Saponification No.	Ether No.	Water reaction.
Colophony, yellow, old	... 167	... 175	... 8	... Clear
„ „ new	... 162	... 169	... 7	... „
„ white	... 172	... 179	... 7	... „

Maly (*Ann.* cxxix., 121 and 389) showed that colophony consisted of 80 per cent. of abietic anhydride, to which he assigned the formula $C_{44}H_{62}O_4$. Therefore the author considers that metallic resinates must consist principally of the metallic derivatives of abietic acid, which is dibasic. The lead compound ($PbC_{44}H_{62}O_5$) contains 23.6 per cent. of lead, whilst the manganese compound ($MnC_{44}H_{62}O_5$) has 7.58 per cent. of manganese.

The 20 per cent. of other colophony acids, sylvic acid ($C_{20}H_{30}O_2$), and its isomer, pimic acid, also combine with lead and manganese to form monobasic salts: $C_{20}H_{20}O_2 \rangle Pb$ (containing 25.6 per cent. of lead), and $C_{20}H_{20}O_2 \rangle Mn$ (with 8.38 per cent. of manganese). Therefore a theoretical lead resinate should consist of 77.1 per cent. of the lead salt of abietic acid, and 22.9 per cent. of the lead salts of sylvic and pimic acid, whilst the proportions of the manganese compounds should be 80.3 and 19.7 per cent. respectively. Calculated on this basis the resinates have the following composition:

Pb	...	24.96	Mn	...	7.74
C	...	59.09	C	...	72.87
H	...	7.10	H	...	8.60
O	...	8.84	O	...	10.78
		<hr/> 99.99			<hr/> 99.99

More recent research (*Mon. Chem.*, 1893, 186) has ascribed to abietic acid the formula $C_{19}H_{28}O_2$, which would give to the lead and manganese salts the formulæ $C_{19}H_{27}O_2 \rangle Pb$, and $C_{19}H_{27}O_2 \rangle Mn$ (containing 26.5 and 8.74 per cent. of their respective metals). Since, however, the question does not appear to the author to be definitely settled, and the difference would not be very great, he has preferred the older formulæ.

In the chemical examination of resinsates the mineral matter (*i.e.*, the metals) and the combined acids should be determined. The author considers that this is most simply accomplished by incinerating a weighed quantity, weighing the oxides, and taking the resin acids by difference; or the resinates may be treated with fuming nitric and concentrated sulphuric acids, and the metals determined in the usual manner. Comparative experiments described show that both methods furnish fairly concordant results.

By direct incineration in a porcelain crucible the subjoined results were obtained:

	Mineral Matter. Per cent.	Organic Matter. Per cent.
1. Resinated Manganese Extract ...	8.9	91.1
2. Manganese Resinate (melted) ...	4.19	95.81
3. " " (precipitated) ...	10.91	89.09
4. Lead and Manganese Resinate (I.) ...	10.4	89.6
5. " " " (II.) ...	12.09	87.91
6. Lead Resinate (melted) ...	20.76	79.24
7. " " (precipitated) ...	25.3	74.7
8. " " (I.) ...	28.47	71.53
9. " " (II.) ...	11.7	88.3
10. " " (III.) ...	29.39	70.61

In many of these samples the proportion of the metal is not what is required by theory. No. 8 contains 4.5 per cent. too much, whilst No. 9 contains 11.3 per cent. too little, or, in other words, 50 per cent. of lead resinate and 50 per cent. of uncombined resin. Nos. 2 and 4 do not contain the right proportions, but Nos. 1, 5, and 7 may be considered pure resinsates. No. 4 was found to contain 8.1 per cent. of lead and 2.3 per cent. of manganese, corresponding to 33.6 per cent. of lead resinate and 30 per cent. of manganese resinate, which leaves 36.4 per cent. of uncombined resin. In No. 5 the amounts were 7 per cent. of lead and 5.23 per cent. of manganese, corresponding to 29 per cent. and 67.5 per cent. of the respective resinsates—a difference of only 3.5 per cent. from the theoretical requirement.

As a further test, the acid and saponification values may be determined. For the acid value, about 2 grammes of the resinate are dissolved in 25 c.c. of a mixture in equal parts of alcohol and ether, and titrated with semi-normal alcoholic potash, phenolphthalein being used as indicator. The saponification number is determined by heating on the water-bath about 2 grammes of resinate with 10 c.c. of alcohol and 25 c.c. of semi-normal potash for half an hour, and titrating back with semi-normal hydrochloric acid. The results obtained with the same samples of resinsates were:

	Acid Value.	Saponification No.
1. Manganese Extract	116	195
2. Manganese Resinate (melted) ...	75	171
3. " " (precipitated) ...	118	185
4. Lead and Manganese Resinate (I.)	75	151
5. " " " (II.)	46	134
6. Lead Resinate " (melted) ...	47	130
7. " " (precipitated) ...	37	128
8. " " (I.) ...	45	126
9. " " (II.) ...	103	165
10. " " (III.) ...	40	128

Although of some value as confirmatory tests, no certain conclusions can be drawn from these results except in the case of No. 9, where the high values show the presence of free resin. This is owing to the resins being decomposed by the alkali. What is required is a solvent which will dissolve colophony readily and leave the resins. The author believes that cold petroleum spirit will do this, and promises a further communication on the subject.

C. A. M.

Estimation of Ethylene in Mixed Gases. P. Fritzsche. (*Zeit. angew. Chem.*, 1896, 456-459.)—In order to estimate ethylene apart from other unsaturated hydrocarbons, the author has made use of the reaction $C_2H_4 + H_2SO_4 = C_2H_6SO_4$, subsequently distilling the ethyl sulphuric acid with water, and determining the amount of alcohol produced. The gas to be examined, after removal of any ammonia or sulphuretted hydrogen, is shaken in the reagent tube with concentrated sulphuric acid, the shaking being continued until, on cautiously opening the tap, no more air enters the tube. The acid is then washed into the distillation flask, distilled, and two-thirds of the distillate distilled after the addition of a little soda. The alcohol in the distillate is determined by taking the specific gravity. The most accurate results are obtained by bringing the alcohol in the fluid to about 1 or 2 per cent.

The process is more tedious where only traces of ethylene are present, and to obtain accurate results with a gas containing only 0.5 per cent. at least 5 litres of gas must be used for the determination, and repeated distillation employed. The author's experiments prove that the alcohol produced corresponds quantitatively with the amount of ethylene present. The absorption-tube employed was 200 millimetres in length and 60 millimetres in diameter, but for use with coal gas its dimensions were 500 by 120 millimetres. It had a capillary tube fitted with a tap at each end. Warming the tube in a water-bath accelerates the reaction. Butylene may be removed from a gas by passing through a wash-bottle containing 70 per cent. sulphuric acid, while any propylene is determined as ethylene.

C. A. M.

The Separation of Trimethylamine from Ammonia. H. Fleck. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 670-672.)—Several chemists have based methods for the detection of trimethylamine in the presence of ammonia on the different solubilities of their hydrochlorides in absolute alcohol. The author shows that when only a low percentage of trimethylamine is present these methods are not even

reliable as qualitative separations. He has found the following quantitative process satisfactory. The mixed hydrochlorides are repeatedly extracted with boiling absolute alcohol, and the solvent distilled off. An excess of sodium hydrate is added to the residue, and the gases formed on boiling driven over into a large quantity of water. This is exactly neutralized with sulphuric acid, litmus being used as indicator. The liquid is evaporated to dryness, and extracted with one litre of cold absolute alcohol, which dissolves trimethylamine sulphate and leaves ammonium sulphate undissolved. After distillation of the alcohol the residue is transferred to a weighed dish, dried, and weighed. In this way 32.910 grammes of the dried mixed chlorides gave 2.5 grammes of trimethylamine sulphate, corresponding to 2.21 grammes of hydrochloride, or 6.71 per cent.

The completeness of the extraction was proved by the total absence of fishy odour on treating the ammonium residue with alkali. The purity of the trimethylamine extract was shown by the analysis of the platinum double salt prepared from the trimethylamine sulphate.

			Per Cent. Pt.	Required for $[\text{N}(\text{CH}_3)_3\text{HCl}]_2\text{PtCl}_4$ Per Cent. Pt.
1.	0.0983	gramme gave ...	36.92	...
2.	0.3017	„ ...	37.12	36.93

C. A. M.

Examination of Commercial Saccharin by means of the Calorimeter. H. Langbein. (*Zeit. angew. Chem.*, 1896, 486-494.)—Saccharin, the anhydride of ortho-sulphamine-benzoic acid, often contains small quantities of para-sulphamine-benzoic acid, which detract from its sweetness. The determination of the melting-point is quite inconclusive, and the author points out that no reliance can be placed on Hefelmann's method, since an error of 0.1 per cent. in the imide nitrogen makes an error of 1.3 per cent. in the amount of saccharin.

For determining the calorimetrical value, a modification of Mahler's bomb calorimeter was employed, and the usual corrections made. With pure saccharin a mean result of 4753.1 calories was obtained, whilst para-sulphamine-benzoic acid gave 4307.3 calories. A mixture of 60 per cent. of saccharin and 40 per cent. of the para-acid gave as the heat of combustion 4574.3 calories, the calculated value being 4574.78 calories. With a mixture containing 2 per cent. of para-acid, the result was 4743.9 calories, corresponding to a calculated quantity of 2.06 per cent.

Owing to the greater solubility of saccharin in various solvents, the para-acid could be concentrated by crystallization and a greater difference obtained. Thus, 11.451 grammes of the above 2 per cent. mixture were dissolved in acetone, and petroleum spirit added until precipitation commenced. There separated 6.1090 grammes, which on combustion gave 4736.1 calories. This corresponded to a calculated percentage of 3.81 per cent. of para-acid in the precipitate, or 2.03 per cent. on the original mixture. With pure saccharin no difference was observed in the heat of combustion of separate portions obtained by fractional crystallization.

Specimens of commercial saccharin examined in this way gave the results here tabulated.:

Saccharin.	Maker.	Sweet-ness compared with Sugar.	M.P.	Moisture per cent.	Ash per cent.	Calories, Ash and moisture-free substance.		Calories, Crystallized fraction.		Para-acid.
						I.	II.	I.	II.	
Sulphinide ...	Heyden	550	224°	0·08	0·098	4754·4	4753·7	4756·2	4754·7	None
Crystalline ...	Heyden	400	—	—	—	4753·6	4755·6	4755·7	—	None
Refined Saccharin ...	Fahlberg and Co.	500	—	0·26	0·06	4749·6	4752·8	4744·8	4746·5	0·37 per cent.
Sucrin ...	Bayer and Co.	550	—	0·19	1·63	4753·2	4756·2	4753·2	4753·6	None
Saccharin ...	Monnet	—	—	0·05	0·04	4752·1	4755·8	4753·3	4754·2	None

C. A. M.

Analysis of the Kola Nut. P. Carles. (*Jour. de Pharm. et Chim.*, 1896, xvi., 104-108.)—For estimation of the alkaloids the author mixes 10 grammes of the finely powdered and sifted nut with 1 gramme of calcium hydrate, adds 20 grammes of 80 per cent. alcohol, and dries until the weight is about 13·5 to 14 grammes. The pulverized mixture is then extracted four times with successive portions (35 c.c.) of a mixture of 100 grammes of chloroform and 20 grammes of alcohol, and the extract dried on the water-bath and weighed. Kola nut, even in extremely fine powder, will not yield all its caffeine to pure chloroform.

Kolanin, the kola red of Heckel, is in the main an insoluble compound of tannic acid with the kola alkaloids. It appears hardly to exist in fresh nuts, since when placed in alcohol they yield little or no coloration. It is produced by the action of a laccase, assisted by atmospheric oxygen, and is not a definite chemical compound, but contains resinous matter, etc. The kolanin from dried Congo nuts contained 10 per cent. of caffeine, that from Indian nuts 16 per cent., and that from Dahomey nuts 20 per cent. Its best solvent is 70 per cent. alcohol. It is insoluble in chloroform, and nearly so in water, but dissolves readily in alkaline solutions, which decompose it. To estimate it, the nut is first extracted with water, and then with 70 per cent. alcohol. The alcoholic extract, washed with cold water and dried at 100° C., gives the amount of crude kolanin. The alkaloids in this are estimated by mixing 1 gramme with 1 gramme of calcium hydrate, 3 grammes of chalk, and 6 grammes of 70 per cent. alcohol, drying the mixture, and extracting with chloroform.

On treating the dried and sifted nuts (200 grammes) with 1 litre of 60 per cent. alcohol for ten days, 1 litre of extract contained in grammes :

	Dahomey Nuts dry.	Dahomey Nuts roasted.	Indian.	Congo.
Dry Extract ...	26·00	18·00	30·00	32·00
Crude Kolanin ...	9·10	4·00	9·50	10·20
Alkaloids ...	3·00	3·00	2·65	2·50

C. A. M.

The Examination of Sumbul Root. J. H. Hahn. (*Amer. Jour. Pharm.*, 1896, lxviii., 395, 396.)—By exhausting the powdered root with petroleum benzine, the author obtained 17·25 per cent. of a fixed oil of a yellowish colour, which became

blackish-brown on keeping. It was thick and viscid, had at first a bland but subsequently a bitter taste, and when rubbed between the fingers gave off a disagreeable odour. It was soluble in alcohol, ether, and carbon bisulphide, and was readily saponified by potash. When a drop of sulphuric acid was added to 3 or 4 drops of oil, a crimson-brown colour was produced, which changed in a short time to dark purple, and after twenty-four hours to brownish-black. On mixing with petroleum benzine and pouring upon a filter, crystals were deposited which could be recrystallized from carbon bisulphide. These were not further examined.

The sample, which was purchased in the open market, contained 4 per cent. of moisture and 8 per cent. of ash.

It has been asserted that much of the sumbul root of commerce is fictitious, but the author was unable to obtain any evidence supporting this statement. The analyses of four samples purchased from different firms all gave the same results.

C. A. M.

The Examination of Powdered Gamboge. E. G. Eberhardt. (*Amer. Journ. Pharm.*, 1896, lxviii., 371-374.)—The test for starch given by the American Pharmacopœia is to boil the sample with water, cool, and test with iodine, when there should be no green colour. This test detects less than 1 per cent. of starch, but in the author's experience it is not easy to find a powdered gum answering to this requirement, although a good pipe gamboge generally does so. It is difficult to account for the presence of the starch, which, being 1 per cent. and less, can hardly be an adulterant. To obtain an idea as to the quantity present, the following test is proposed: One gramme of the powdered gamboge is dissolved in 5 c.c. of caustic potash solution, 45 c.c. of distilled water added, and an excess of hydrochloric acid, the whole being stirred until of a uniform yellow colour. The liquid is then strained through a plug of cotton-wool, placed in the neck of a funnel, and the almost colorless liquid tested with iodine solution. If more than 2 per cent. of starch be present, a dark colour or precipitate is immediately produced. Commercial gamboge gives a greenish tint, gradually developing into faint blue. Pure gamboge, with 1 per cent. of starch or flour, gives a faint blue, darkening on standing, and depositing a slight precipitate. Two per cent. of starch gives an immediate decided blue, while 5 per cent. gives a precipitate almost immediately. Five per cent. and less of curcuma gives a clear starch reaction.

But freedom from starch does not necessarily show purity, and the author considers the estimation of the resin, which is the active part of the gamboge, as the only proper criterion. This should not be less than from 75 to 80 per cent. In the following table are given the amounts of resin extracted from different varieties of air-dried gamboge by alcohol:

				Pipe.	Pipe.	Pipe.	Powdered.
Resin	78.9	76.54	75.9	81.4
Starch	none	none	none	trace

C. A. M.

On Kjeldahl's Method and its Modifications. G. Rivière and G. Bailhache. (*Bull. Soc. Chim.*, xvi., 806-811.)—This paper contains an account of experiments made by the authors with the object of shortening the time required for decolorization with sulphuric acid. Their chief objection to mercury is the necessity of subsequent precipitation, and the inaccuracy introduced if too much be used. With phosphoric acid in place of sulphuric acid the results were not satisfactory, even after the addition of manganese dioxide and phosphate of manganese. By adding ferric oxide to the phosphoric acid correct results were obtained, but the process was slower and more unpleasant than Kjeldahl's method.

Various substances were tried as means of raising the temperature of the sulphuric acid, such as iron sulphate, vanadic acid, molybdic acid, boric acid, and arsenic acid; but in each case where the results were correct the process was too slow.

The only objection the authors have to the use of potassium bisulphate is that with certain substances, such as horn, a relatively long time is required to complete the conversion.

The modification found to give the most speedy results was the addition of sodium pyrophosphate, obtained by calcining the ordinary phosphate. At the same time a smaller amount (1 to 2 grammes) was required than in the case of potassium bisulphate.

The following comparative results of the time required for decolorization and the percentage of nitrogen obtained are taken from the long table given in the original paper :

Substance.	Soda lime combustion.	Kjeldahl with mercury.	Addition of Potassium Sulphate.				Addition of Sodium Pyrophosphate.	
			With 10 grammes. 1 to 2 hours.	With 5 grammes. 2 to 3 hours.	With 2 grammes. 2½ to 3½ hours.	With 1 gramme. 3 to 4½ hours.	With 2 grammes. 1 to 2 hours.	With 1 gramme.
Horn	13:00	13:00	13:15	13:15	13:20	13:20	13:20	13:20
Flesh	10:34	10:35	10:41	10:45	10:45	10:45	10:40	10:45
Blood (dried)	10:45	10:45	10:55	10:55	10:60	10:65	10:65	10:60

Prior knowledge of Dr. Dyer's work on this subject is disclaimed.

C. A. M.

The Chemical Nature of Diastase II. T. B. Osborne and G. F. Campbell. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 536-542.)—The results of the former investigation (*ANALYST*, xx. 232) have been confirmed, but no diastase with greater activity has been obtained. The method employed was the same as before, the malt (15 kilos.) being extracted with sodium chloride brine, the filtrate saturated with ammonium sulphate, the precipitate dialyzed in water to remove globulin, and the solution dialyzed into alcohol of varying strengths, commencing with alcohol of 0.86 specific gravity, and ending with absolute alcohol, the various precipitates being filtered off. The six fractional precipitates thus obtained were treated with water, in which the two last were completely soluble, the others partially so. The filtered aqueous solutions were dialyzed first into water and then into alcohol, and absolute

alcohol finally added until the proteids were completely precipitated. The six preparations, which weighed (a) 1.37, (b) 1.47, (c) 4.05, (d) 4.82, (e) 2.17, and (f) 0.63 grammes respectively, were dehydrated with alcohol, and dried over sulphuric acid. Their diastatic capacity, determined in the same way as before, was (a) = 0, (b) = 60, (c) = 300, (d) = 300, (e) = slight, (f) = 0.

Fractions c and d, which contained nearly all the enzyme, were united, dissolved in water, and filtered from insoluble matter, which, when washed with water and absolute alcohol and dried over sulphuric acid, weighed 0.23 gramme. The filtrate and washings were treated with alcohol, which made a solution containing 36.5 per cent. of alcohol. The precipitate (0.25 gramme) had a diastatic power of 15. The filtrate from this was treated with more alcohol, raising the percentage to 50.7. The precipitate weighed 2.35 grammes, and had a diastatic capacity of 86. The alcohol in the filtrate was brought to 61.6 per cent., and the resulting precipitate weighed 2.87 grammes, and had a diastatic power of 600. Absolute alcohol was added to the filtrate, and gave a precipitate (1.00 gramme) with a diastatic value of 100. When more absolute alcohol was added to the filtrate from this, a precipitate was produced which weighed 0.4 gramme, and had only a trace of diastatic power. More absolute alcohol added to the resulting filtrate gave 0.17 gramme of substance which was totally inactive; and, finally, the filtrate from this, when evaporated to dryness, left a residue weighing 0.65 gramme.

These results show that nearly all the diastase was thrown down when the proportion of alcohol in the solution was brought to about 60 per cent. The diastatic power of 600, which was also the highest obtained in the former investigation, could not be exceeded. An attempt was made to further purify the most active preparation by treatment with water and absolute alcohol, but the resulting substance lost one-third of its diastatic activity in the process. A similar series of experiments were made with a diastatic malt extract, but no diastase could be obtained with greater power than 300.

The authors consider that diastatic activity is largely dependent on the presence of other substances, and that the purer the diastase becomes the more sensitive it is to external conditions, and hence the determination of its maltose-producing power may not necessarily be the most certain test for its purity. In support of this view it is pointed out that the addition of sodium chloride often promotes diastatic activity, whilst it is probable that albumen is an essential factor, since in this investigation those preparations which contained the most albumen were always found to be the most active, and in no case was diastatic activity exhibited in the absence of albumen.

W. J. S. and C. A. M.

INORGANIC ANALYSIS.

Electrolytic Determination of Lead, and the Influence of Arsenic, Selenium, and Manganese thereon. B. Neumann. (*Chem. Zeit.*, xx., 1896, 381.)—Although lead can be deposited from a large number of solutions by electrical agency either as metal or peroxide, it is only in the latter form that it is available for quantitative

analysis. In nitric acid solution, provided the liquid contains at least 10 per cent. of free acid, the lead is completely thrown down as peroxide, the current being preferably 1 to 2 ampères per 100 square centimetres of anode surface at a tension of 2.3 to 2.7 volts. The operation may be conducted either at ordinary temperatures or at 60° to 70° C., and the anode should be a platinum basin with a matt surface. As the oxide contains water, it must be dried at 180°. In the presence of other metals Zn, Fe, Co, Ni, Al, and Mn remain in solution; while Cu, Sb, Au, and Hg are deposited with the lead. Ag and Bi may be thrown down partly as metal and partly as oxide; but if they are present only in comparatively small amount, and the free acid be increased to 20 per cent., the error becomes too small to be appreciable. For the analysis of lead and copper ores, brass, bronze, etc., the electrolytic process is very exact and convenient, and even extremely small quantities of lead can be determined by its help.

The author has carried out a large number of experiments on the influence of arsenic, selenium, and manganese on the electrolysis of lead salts, using solutions prepared so as to imitate the conditions obtaining when 1 gramme of an ore with 22.38 per cent. of lead and the impurities in question should be under analysis, and his conclusions may be briefly summarized as follows: Although it is possible to recover the whole of the lead by the use of a large excess of acid, and by allowing the current (0.5 to 0.6 ampère per square centimetre) to pass for a long time, arsenic or selenium in any quantity above 0.5 per cent. on the ore practically prohibits the use of the method. Providing manganese is not present in excess of 3 per cent., that the electrolysis is conducted at 70° with a fairly strong current (1 to 1.3 ampère per 100 square centimetres at 2.5 to 2.7 volts) passing for not over thirty minutes, and that sufficient acid (30 c.c. of 1:4 nitric per gramme of ore) is employed, the results may be considered reliable. An experiment where 0.5 per cent. each of As and Se and 1 per cent. of Mn were present at the same time was fairly successful, but would probably not have been so had the amounts of the impurities been higher.

F. H. L.

Volumetric Estimation of Lead. F. J. Pope. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 737-740.)—The chief objection to the various processes which have been proposed is the use of an outside indicator. To obviate this, the author describes a method in which the lead is first converted into sulphate, and then into acetate. An excess of standard potassium bichromate is added, which precipitates the lead as chromate; the unused bichromate is reduced by excess of standard arsenious acid, and this titrated with iodine solution.

The solutions required are:

Iodine.—12.7 grammes dissolved in concentrated potassium iodide solution and made up to a litre.

Arsenious Acid.—4.95 grammes dissolved in 20 to 30 c.c. of saturated filtered solution of sodium carbonate with gentle warming, and made up to a litre. This is standardized by slightly acidifying 10 to 15 c.c. with sulphuric acid, adding 50 c.c. of saturated solution of pure sodium bicarbonate, and titrating with iodine, starch

paste being used as indicator. The iodine value of the bicarbonate used should be determined, and a correction made.

Potassium Bichromate.—Five grammes per litre, standardized on the arsenious acid solution.

From 3 to 7 grammes of the ore are moistened with water in a porcelain dish, treated with 4 or 5 c.c. of a mixture of two volumes of sulphuric acid, three volumes of nitric acid, and one of water, and evaporated as nearly as possible to dryness without spurning. After cooling, water is added, the precipitate filtered off, and washed until nearly free from acid. The filter-paper and precipitate are placed in a 300 to 400 c.c. beaker, any remaining acid neutralized with dilute ammonia, and the whole boiled for about ten minutes, with constant stirring, with 10 to 15 c.c. of strong ammonium acetate solution acidified with acetic acid. After cooling, the liquid is neutralized with ammonia, and 100 c.c. of standard bichromate solution added. The precipitate is removed by filtration, washed with dilute ammonia or ammonium acetate, and the filtrate made up to 500 c.c. To 100 c.c. of this solution in a 1,500 c.c. basin from 10 to 20 c.c. of standard arsenious acid solution are added, followed by sulphuric acid (40 per cent.) until decidedly acid, a large excess being avoided. The liquid is stirred until it assumes a greenish tint, or until the yellow colour disappears. It is then neutralized with a saturated solution of sodium bicarbonate, and an excess of 50 c.c. added, the solution being diluted with water if it has a greenish tint. Finally, starch paste is added, and the liquid titrated with standard iodine solution.

With a specimen of galena containing quartz and calcium carbonate, the percentage of lead found by the author in four determinations varied from 81·89 to 81·96. With another ore containing 5 per cent. of copper, 26 per cent. of iron, quartz, and gypsum, another operator found (1) 15·89, (2) 16·01, and (3) 15·97 per cent. of lead.

With pure lead sulphate the following results were obtained :

Grammes taken.							Grammes found.
1·0	1·000568
1·1	1·099375
1·2	1·200467
1·3	1·300673
1·4	1·399571

C. A. M.

Analysis of Aluminium and its Alloys. H. Gouthière. (*Ann. de Chimie. Analyt.*, i. [14], 265-270.)—*Copper, Nickel, Lead, and Iron.*—From 5 to 10 grammes of aluminium turnings are attacked in successive portions by sodium hydroxide solution (1 to 3) in a conical 300 c.c. flask. Heat is evolved, brisk effervescence occurs, and the operation is complete in about twenty minutes, care being taken to prevent the liquid cooling, or a gray deposit, difficult to dissolve, will be formed. The hot, clear liquid is decanted on to a small filter, and the black deposit very quickly washed five or six times with boiling water, after which it is dissolved in luke-warm dilute nitric acid, evaporated to a syrup, mixed with 2 c.c.

of pure nitric acid, and electrolysed by the current from three or four Leclanché cells at a temperature of 50° to 60° C. Both electrodes are of platinum, the negative being a crucible or cylinder, and the positive preferably gauze. In two or three hours all the copper will be deposited as metal on the negative electrode and the lead on the positive as PbO_2 , the weight of which, multiplied by 0.8661, will give the metallic lead.

The liquid separated from the copper and lead is united with the washings, and heated with excess of ammonia to throw down the iron and dissolve the nickel precipitate first formed. When the proportion of the latter metal is large, the iron precipitate should be redissolved in dilute nitric acid and reprecipitated as before. After filtering and washing with boiling water, the iron oxide is dried, calcined, and weighed. The ammoniacal filtrate is heated to drive off the excess of ammonia, then mixed with a few drops of sulphuric acid, concentrated, nearly neutralized by ammonia, and then electrolyzed at about 60° C., the nickel depositing in the metallic state on the negative electrode.

Aluminium is generally estimated by difference, on account of the time and trouble required by the washings and incineration. If determined direct, one of the usual methods, or that described by Moissan (*Ann. de Chimie Analyt.*, 1896, p. 10), may be followed.

Tin, Antimony.—Five or ten grammes of aluminium are attacked by hydrochloric acid, and, after nearly neutralizing the solution by ammonium carbonate, a current of hydrogen sulphide is passed, the resulting precipitate being collected, washed with hydrogen sulphide water, and digested for some time with dilute ammonium sulphide at a moderate heat. Then, after filtering and washing with ammonium sulphide water, the soluble sulphides are thrown down by a slight excess of dilute hydrochloric acid, or, better, by the aid of heat. Sulphur is removed by carbon bisulphide; antimony (which is rarely present) is recognised by the colour of the sulphide; arsenic sulphide volatilizes during calcination, and stannic oxide remains for weighing.

Zinc is precipitated from the hydrochloric acid solution (after removal of the tin, antimony, etc.) by hydrogen sulphide following an addition of sodium acetate. The precipitate—sometimes containing sulphur—is washed, calcined, and weighed as ZnO .

Arsenic.—The sulphides precipitated from 5 grammes of the substance are washed and dissolved in dilute nitric acid. The solution is filtered, evaporated with sulphuric acid until white fumes appear, and then placed in a Marsh apparatus. The gas evolved is passed through a solution of silver nitrate, and the precipitated silver converted into, and weighed as, silver chloride. This, multiplied by 0.0871, gives the arsenic.

Silica.—The solution resulting from the action of hydrochloric acid and 3 or 4 c.c. of nitric acid on 5 grammes of aluminium is evaporated to dryness until the acid is driven off; the mass is then heated to 110°–120° C., taken up with hot dilute hydrochloric acid, digested for awhile, and, after adding boiling water and pouring off the liquid, washed with boiling water, dried and calcined in a muffle.

The dry chlorine method may be also employed, but necessitates a special and complex apparatus.

Total Carbon. — Boussingault's method is used—chlorination by mercuric chloride, digestion in dilute hydrochloric acid, and heating in a current of hydrogen. Creak and Ulgreen's method is also applicable—solution of the aluminium in cupro-ammonium chloride, removal of the precipitated copper by hydrochloric acid, and combustion of the carbon by a mixture of chromic and sulphuric acids in a special apparatus provided with potash absorption-tubes.

Sulphur is estimated by the Rollet method—2 grammes of aluminium being heated to redness, in a current of two-thirds hydrogen and one-third carbon dioxide, and the hydrogen sulphide passed into silver nitrate solution. The silver sulphide is washed and calcined, the silver multiplied by 0.1481 giving the weight of sulphur. A fairly accurate estimate of the sulphur may be rapidly made by the Eggertz colorimetric method, noting the effect of the hydrogen sulphide on a silver plate.

The results of ten analyses of aluminium are given, the chief impurities being lead, iron, and copper; tin, arsenic, and nickel were only found in two samples.

C. S.

Determination of Iron Oxide and Alumina in Phosphate Rock by the Ammonium Acetate Method. T. S. Gladding. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 717-721.)—In spite of the adverse criticisms which have been passed upon this method, the author describes experiments to prove that, when carried out under the right conditions, it gives an accurate separation of iron and alumina from calcium phosphate, and yields a neutral phosphate, in which the two constituents may be accurately determined. He shows that when iron and aluminium are repeatedly precipitated as phosphates, there is no appreciable loss, provided a large excess of phosphoric acid be present. Moreover, in the presence of a large amount of calcium phosphate, three successive precipitations leave the iron and aluminium phosphates sufficiently pure to be estimated as such.

The process recommended is as follows: Four grammes of the finely-ground sample are digested for 30 minutes at a temperature just below boiling with about 30 c.c. of dilute hydrochloric acid (1 : 1), which prevents the solution of any pyrites present. The solution is filtered and washed into a 200 c.c. flask, oxidized with nitric acid and made up to the mark. Two portions are taken—50 c.c. = 1 gramme, and 25 c.c. = $\frac{1}{2}$ gramme. These are nearly neutralized with strong ammonium hydrate, thoroughly cooled, and the neutralization completed with dilute ammonium hydrate until the precipitate just remains permanent; dilute hydrochloric is then added until the precipitate just dissolves. A mixture of 15 c.c. of strong ammonium acetate solution (made by neutralizing 30 per cent. acetic acid with strong ammonia) and 5 c.c. of acetic acid is poured into the beaker in a thin stream, the liquid being meanwhile stirred. After digesting for 30 minutes to an hour at 60° C., the precipitate is filtered off and the beaker washed with ammonium acetate solution (10 per cent.). The precipitate is dissolved from the paper with a few c.c. of hot dilute hydrochloric acid (1 : 4). One gramme of ammonium phosphate is added, and the liquid neutralized with ammonium hydrate until the precipitate just dissolves as before, and poured

into a mixture of 15 c.c. of ammonium acetate solution and 5 c.c. of acetic acid. The digestion, filtration, etc., are again repeated to this point, and the precipitate washed three times with dilute ammonium acetate solution.

The filter while wet is ignited in a platinum crucible, finishing with a blast, and the combined phosphates of iron and aluminium weighed together. The iron is determined volumetrically in the solution of the weighed precipitates. The iron oxide in the rock is also estimated volumetrically, preferably by the bichromate method.

C. A. M.

A New Method of Estimating Iron Oxide and Alumina in Phosphate Rock.

T. S. Gladding. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 721-724).—To obviate the necessity of the repeated precipitations in the ammonium acetate method (see preceding abstract) the following modification is described. It consists in the separation of alumina from calcium phosphate and iron oxide by means of its solubility in caustic potash. Four grammes of the finely pulverized rock are dissolved in dilute hydrochloric acid (1 : 1), heated for 30 minutes just below the boiling-point. The solution is filtered into a 200 c.c. flask, oxidized with nitric acid, cooled and diluted to the mark. To 50 c.c. of the solution, 25 c.c. of pure caustic potash solution (500 grammes per litre) are added, the whole digested for an hour at 70° C., and the precipitate filtered off and washed with hot water.

One gramme of ammonium phosphate is added to the filtrate, then hydrochloric acid to acid reaction, and ammonia until a permanent precipitate is formed. This is just dissolved in hydrochloric acid, ammonium acetate solution and acetic acid added as described in the preceding abstract, and the contents of the beaker digested at 70° C. for an hour. The precipitate is filtered off, washed five or six times with 10 per cent. ammonium acetate solution, ignited, and weighed as normal aluminium phosphate.

The iron oxide is determined volumetrically in a solution of the precipitate of iron oxide and calcium phosphate, thrown down by the caustic potash.

The results obtained by this method are in close agreement with those given by the ammonium acetate process.

C. A. M.

Reactions of Vanadic and Molybdic Acids with Thiocyanates. **W. Ellram.**

(*Proceedings of the University of Dorpat*, 1895, xi., 28; through *Chem. Zeit. Rep.*, 1896, 153).—If powdered ammonium vanadate and one drop of sulphuric acid are added to the solution of a thiocyanate, the liquid turns yellow; but if the acid is increased to 5 or 15 drops it alters to sky-blue, and is unchanged by further additions or by boiling. If some of the vanadate still remains undissolved, the colour returns to yellow in the presence of more acid. The reaction will detect 1 part of a thiocyanate in 12,000. It is available in the case of saliva, but here the colour has a somewhat greenish cast, and it is rendered cloudy by albumen. In serum the latter substance must first be removed with zinc sulphate. In milk the test also succeeds, but all animal liquids need to be purified as much as possible. Conversely, vanadic acid or its salts may be detected by means of a thiocyanate, even in dilutions of

1 part per 5000: a few crystals of the reagent being added to the liquid and then sulphuric acid till the yellow colour changes to blue.

Molybdic acid and its salts give with potassium thiocyanate a yellow, orange, or blood-red colour, visible after a minute in dilutions of 1 per million. With zinc and sulphuric or hydrochloric acid molybdates yield an indigo-blue colour, or a green, either olive, grass, or pale in shade. The intensity depends on the concentration, and with excess of metal the colour is usually blue, changing into the greens on further additions of acid. The reaction is available up to 1 part per 35,000.

F. H. L.

Estimation of Dissolved Oxygen in Water. Romija. (*Rec. trav. chim. des Pays-Bas*, 1896, xv., 76; through *Chem. Zeit. Rep.*, 1896, 191.)—A gas pipette fitted with stop-cocks at either end, and provided with a funnel holding about 1 c.c. on the top, is filled with the sample of water. One c.c. each of solutions containing respectively (1) 1.12 gramme of manganese chloride and 0.085 gramme of potassium iodide, (2) 10 grammes of Rochelle salt, and (3) 0.1 gramme of caustic soda per 12 c.c., are introduced successively by opening the taps, and the tube is well shaken and allowed to stand for ten minutes. One c.c. of 25 per cent. hydrochloric acid is then added, and the whole rinsed out into a flask, and the liberated iodine titrated with thiosulphate. An allowance for the 4 c.c. of water displaced by the reagents must be made.

F. H. L.

Analysis of Crude Monazite, and the Estimation of Thorium, etc. C. Glaser. (*Chem. Zeit.*, 1896, xx., 612.)—Since the advent of the "incandescent" system of gas-lighting, monazite has become an article of commerce; and the older methods for the valuation of the native mineral, which consisted in determining the ferric oxide, silica, and titanitic acid, and returning the residue as a pure phosphate of cerium, etc., are no longer sufficiently exact. The author has checked the statements made by different investigators respecting the behaviour of the rare earths with various reagents, and his observations are shown in the accompanying table. He has also conducted a large number of experiments on the separation of these substances one from another (for full details of which the original paper must be consulted), and has embodied his results in the following scheme for the analysis of monazite.

After being finely powdered, the sample is dissolved by boiling for some time in strong sulphuric acid, any insoluble residue being attacked by fusion with potassium bisulphate; if it be desired to estimate the silica, however, it is better to avoid the latter process, and to break up the mineral simply by repeated digestion in acid. The mixture is then poured into ice-cold water; everything, except the silica, tantalic acid, and possibly some titanium and thorium, is in the solution. The residue is heated twice with hydrofluoric acid, moistened with sulphuric acid, dried, and ignited, the silica being found from the loss in weight. If anything remains after this treatment, it is boiled with sulphuric acid or melted with bisulphate, the undissolved matter being returned as tantalic acid, and the solution mixed with the original one. Sulphuretted hydrogen is then passed through the hot liquid until it becomes cold, causing the precipitation of titanitic acid and the metals of the fifth

group. When these have settled, they are filtered off and washed, the filtrate boiled till free from H_2S , the greater part of the free acid neutralized by ammonia, 100 c.c. of a cold-saturated boiling solution of ammonium oxalate for every 2 grammes of the sample added, and the whole allowed to stand over night. The precipitate contains the oxalates of the cerium earths and thorium, and the solution H_3PO_4 , Fe, Al, Mn, Be, Y, Zr, and Ca.

To separate the latter, they are thrown down by ammonia, washed, ignited, and fused with sodium potassium carbonate. The melt is extracted with water, the liquid mixed with the previous filtrate, and the phosphoric acid and the alumina estimated in aliquot portions. The insoluble part of the melt is dissolved in H_2SO_4 , precipitated with NH_3 , and the lime determined in the filtrate. The paper containing the residue is burnt, the ash dissolved in warm HCl , and almost neutralized with NH_3 .

Excess of a solution of ammonium carbonate with sufficient sulphide to throw down the metals of the fourth group is then placed in a beaker, and the last-mentioned solution poured in. The iron and manganese are precipitated, and estimated as usual; the filtrate containing Zr, Yb, and Be, is boiled for an hour, when these bodies are completely precipitated. This precipitate is dissolved in HCl , boiled, cooled, and an excess of NaHO added. The two former earths fall; the Be remains in solution, and can be recovered by boiling the liquid. To separate the Zr and Yb, the hydrates are dissolved again in HCl , the solution warmed and saturated with potassium sulphate, allowed to cool, and the insoluble double salt of Zr filtered off. The Yb may be precipitated from the filtrate by ammonia.

The oxalates of the Ce metals are ignited, the resulting oxides dissolved in H_2SO_4 , the liquid nearly neutralized with NH_3 , heated to the boil, and an excess of a boiling solution of ammonium oxalate added. After a few minutes, when the oxalates of the Ce group have formed again, a few c.c. of ammonium acetate are introduced, and the whole allowed to cool. Next day the liquid is filtered, and the Th precipitated from the filtrate by NH_3 . The Ce may be separated from the La and Di by leading chlorine through the freshly-precipitated hydrates in the usual manner.

Treated in this way, three samples of monazite from North Carolina gave:

	I.	II.	III.
SiO_2	6.40	3.20	1.45
TiO_2	4.67	0.61	1.40
Ta_2O_5	0.66	—	6.39
P_2O_5	18.38	28.16	26.05
Pb	trace	—	—
Al_2O_3	1.62	absent	0.15
CaO	1.20	„	—
CeO	32.93	63.80	59.09
La_2O_3 and Di_2O_3	7.93	2.32	1.19
ThO_2	1.43	absent	0.65
Fe_2O_3	7.83	trace	—
Mn_2O_4	—	—	—
ZrO_2 and YbO (?)	13.98	1.52	2.68
Be_2O_3	1.25	—	—
	98.28	99.61	99.05

BEHAVIOUR OF THE EARTHS WITH DIFFERENT REAGENTS.

	K or NaHO.	K ₂ or Na ₂ CO ₃ .	K ₂ or Na ₂ SO ₄ .	Na ₂ S ₂ O ₃ .	NH ₃ .	(NH ₄) ₂ CO ₃ .	(NH ₄) ₂ S.	NH ₄ Cl.	(NH ₄) ₂ C ₂ O ₄ .	NH ₄ C ₂ H ₃ O ₆ .
Al ₂ O ₃ ...	ppt. sol. in excess	ppt.	form. of alum	ppt. in neutral sols. on boiling.	ppt.	ppt.	ppt. hydrate	sol. in NaHO pptd.	no ppt.	ppt. on boiling sol. of basic salt
Be ₂ O ₃ or BeO ...	ppt. sol. in excess, re-pptd. on boiling or dilution	ppt. sol. in large excess, or by addition of CO ₂	no ppt., or double salt	no ppt.	ppt. insol. in excess	ppt. sol. in excess, re-pptd. on boiling, Al ₂ O ₃ interferes	ppt. hydrate	hydrate sol. on long boiling, NH ₃ given off	no ppt., double salt formed.	(?)
ZnO ₂ ...	ppt. insol. in excess	ppt. slightly sol. in excess, pptd. by NH ₃	double salt if hot, pptd. on cooling in sol. in HCl.	ppt. hydrate and sulphide	ppt. insol. in excess	ppt. sol. in large excess, re-pptd. on boiling	ppt. hydrate	no ppt.	ppt. sol. in excess.	(?)
Gadolinite earths ...	ppt. insol. in excess	ppt. sol. in excess, insol. in double salt (Y), pptd. after a time	ppt. in strong sol. in H ₂ O & K ₂ SO ₄ , Y not pptd.	(?)	ppt. insol. in excess	ppt. sol. in sol. double salt pptd. (Y)	ppt. hydrate	(?)	E pptd. from hot acid sol. as red powder.	(?)
ThO ₂ ...	ppt. insol. in excess	ppt. sol. in NH ₃ gives ppt. on heating, sol. again in the cold	insol. double salt with K. Na salt more sol.	imperfectly pptd. even on boiling strong sols.	ppt. insol. in excess, falls before cerite	as with NH ₃	as with NH ₃	no ppt.	ppt. sol. in excess hot, ppt. on cooling	no ppt., hinders the pptn. with (NH ₄) ₂ C ₂ O ₄ ; from this sol. partly pptd. by HCl, or as hydrate by NH ₃
CeO ...	ppt. insol. in excess	ppt. slightly sol.	double salt insol. in K ₂ SO ₄	no ppt.	ppt. insol., after ThO ₂	as with NH ₃	as with NH ₃	no ppt.	ppt. even in acid sol., in strong acid ditto	does not hinder pptn.
Ce ₂ O ₃ ...	ppt. flesh-coloured by excess	ppt. almost insol.	—	no ppt.	as KHO	ppt. sol. in large excess, pptd. on boiling	as with NH ₃	no ppt.	does not hinder pptn.	does not hinder pptn.
La ₂ O ₃ ...	ppt. insol. in excess	ppt. almost insol.	insol. double salt	no ppt.	as KHO	ppt. insol. in excess	as with NH ₃	no ppt.	ppt. in neut. or ammon. sol. (?)	does not hinder pptn.
Di ₂ O ₃ ...	ppt. insol. in excess	ppt. almost insol.	insol. double salt	no ppt.	as KHO	red ppt. in sol.	as with NH ₃	no ppt.	ppt. white to pale-red, almost insol. in dilute acid	does not hinder pptn.

The samples were cleaned from rutile, granite, and other impurities before analysis.

F. H. L.

NOTE.—This paper contains a good many statements of doubtful accuracy, and the method in its general course is far from the simplest that can be adopted for the separation of the rare earths into fairly well-defined groups.

O. H.

APPARATUS.

Litmus Pencil.—This useful little appliance, which has recently been introduced by Messrs. Christy and Co., of London, has the form of an ordinary lead-pencil, one half of its core being filled with blue, the other half with red litmus. In using it, the ends are sharpened and lines drawn on a strip of white neutral paper with one of the slightly-moistened points. The lines thus produced are said to be much more sensitive than ordinary litmus-paper.

W. J. S.

REVIEWS.

NITRO-EXPLOSIVES. By P. GERALD SANFORD, F.I.C., F.C.S. London: Lockwood and Co. Price 9s.

THIS small volume gives much useful and interesting information, and, with a little more care on the part of the author, might have been a valuable addition to our libraries.

As it is, the book suffers somewhat from imperfect arrangement, leading, among other drawbacks, to unnecessary repetitions. In parts either too much or too little detail is given, and there are various inaccuracies and omissions of what, to English chemists at least, would be important information.

Thus the author deals with blasting gelatine, gelatine dynamite, cordite, etc., before describing the manufacture and preparation of the various nitro-celluloses. The proper arrangement would have been to give the manufacture and properties of all the various nitro-compounds used in the preparation of explosives before treating these explosives themselves.

In the next place, it appears to us that the part dealing with the manufacture of nitro-glycerine, nitro-cellulose, etc., while giving details not necessary to the general chemist, is yet not detailed enough to serve as a really useful guide to the manufacturer.

Further, the author does not bring out sufficiently clearly the very marked differences existing between the two chief classes of so-called nitro-explosives, viz., the nitric ethers, like nitro-glycerine and nitro-cellulose and similar compounds, on the one hand, and true nitro substitution compounds, like tri-nitro-phenol, mono- and di-nitro-benzole, etc., on the other. He thus misses the chance of bringing home to manufacturers and inventors some valuable lessons based on this difference. Nitric ethers, although stable at ordinary temperatures when perfectly pure, are far more prone to decomposition at slightly elevated temperatures than the true nitro compounds. This difference is still more strikingly manifested in the presence of free acids. Hence the absolute necessity, not only of freeing the nitric ethers from all

acids, but also of not bringing them into contact with substances liable to become acid. Nitrate of ammonium, for example, is liable to lose ammonia and become acid. This change is of little or no importance when the nitrate is mixed with a true nitro-compound, but its presence is fatal to the stability of any nitric ether. Hence explosives like bellite, roburite, etc., are unobjectionable as regards their stability, whereas the admixture of this salt with gun-cotton, nitro-glycerine, etc., is inadmissible, and no explosive of the latter class is licensed in this country.

It is also a pity that the author has not extended his study of official documents a little more widely than he seems to have done, as he would then have been saved from several errors. Thus, had he examined the list of explosives authorized to be manufactured in, or imported into, this country, he would have learned that roburite is not a mixture of nitrate of ammonium, and chloro-di-nitrobenzole, but a mixture of the former salt with chlorinated di-nitrobenzole, containing, at most, 4 per cent. of chlorine, corresponding, roughly, with a mixture of 1 part of chloro-di-nitrobenzole and 3 parts of di-nitrobenzole. He would also have seen that there is no such explosive as his so-called Rhenish dynamite legally in use in this country; can the author have mistaken dynamite manufactured by the Rhenish Dynamite Company for his Rhenish dynamite? As a matter of fact, out of the thirty-four explosives given at the end of the work as being in common use, sixteen only are authorized in this country. Surely in a work intended primarily, we suppose, for use in England, such an important fact should have been mentioned. Most explosives given in this table not only are not authorized in this country, but the composition of several, as given, is incompatible with their stability; as, for example, ammonia nitrate powder, which is stated to contain nitrate of ammonium and chlorate of potassium, two salts incompatible with each other; or his ammonium dynamite, containing nitro-glycerine and nitrate of ammonium, which, as before stated, are also incompatible with each other. Again, blasting gelatine with only 3 per cent. of nitro-cotton would never be allowed to be issued by itself; carbonite, as authorized, has not the composition given. Ordinary dynamite does not usually consist of 25 parts of kieselguhr and 75 parts of nitro-glycerine; usually it contains only from 73 to 74 per cent. of nitro-glycerine, and the above-given proportion is simply the maximum proportion of nitro-glycerine permitted by law; it is, so to speak, the ideal, but not the actual, composition.

We also cannot help thinking that the author should have raised some note of warning against the use of chlorate of potassium. Chlorate of potassium offers great temptations to the inventor of explosives. There are, however, many drawbacks connected with its use, such as great sensitiveness of the resulting explosive to percussion and friction, and great liability to spontaneous decomposition. These difficulties have, up to the present, proved practically insurmountable, and no chlorate-explosive, in which this salt forms an essential constituent, is licensed in this country.

The chapter on the estimation of nitrogen is well done and fairly complete; many of the other parts on the analysis of explosives, however, require revision. Has the author never tried to dissolve the nitro-glycerine from dynamite by means of methylic alcohol? This alcohol, although by far the best solvent for nitro-glycerine, is, strangely enough, not even mentioned among the solvents given on page 19. We certainly know of no chemist accustomed to the analysis of blasting gelatine, cordite,

etc., who estimates the nitro-cotton contained in these explosives by means of the method given by the author, viz., precipitation with chloroform. We should also like to ask the author whether he has ever tried to estimate the moisture in a sample of wet gun-cotton by drying the same in a water oven at 100° C.? We can only warn our readers against making the attempt if they wish to obtain accurate results. We hope that, in a second edition, the author will deal with some, at least, of the points raised above, and supply us with a much-wanted manual at once complete and accurate. Such a book, of moderate size, would be a most valuable addition to the library of every practical chemist.

A. D.

A HANDBOOK OF INDUSTRIAL ORGANIC CHEMISTRY. By SAMUEL P. SADTLER, Ph.D., F.C.S., Professor of Chemistry in the Philadelphia College of Pharmacy, and in the Franklin Institute of the State of Pennsylvania. Second enlarged and revised edition. Philadelphia and London: J. B. Lippincott and Co., 1895.

We are glad to see that the excellent "Handbook of Industrial Organic Chemistry" published by Dr. Sadtler in 1891 has already reached a second edition. The work compares very favourably with some so-called technical manuals published in this country, and shows that the author is fully alive to the requirements of the day. The present edition extends to 537 pages, including the index, against 519 pages in the case of the first edition. In his preface Dr. Sadtler points out that the bibliography has in all cases been rewritten and brought carefully to date, and the statistics have also been brought down to 1895 whenever new figures were obtainable.

While the body of the text has not been materially altered, numerous corrections have been made and new sections inserted in many cases. In the appendix two new tables have been added, giving the physical and chemical constants of the oils, fats, and waxes, classified for correction and comparison. The arrangement of the work appears to be well adapted for the convenience of the user of the book.

Referring to particular sections, we are sorry to see that in that on vinegar no addition has been made to the somewhat bald article of the first edition; and no reference is made to the information on the subject of vinegar published within the last few years in the pages of the ANALYST. The section on flour-testing might also be revised with advantage.

These are minor defects; and Dr. Sadtler is to be congratulated on the ability and thoroughness with which he has treated such varied and numerous subjects within a reasonable space. The work is eminently one for reference, and should find a place in the laboratory of every analyst and manufacturing chemist. The type is everything that can be desired, and the illustrations are satisfactory.

A. H. A.

COMMERCIAL ORGANIC ANALYSIS. By ALFRED H. ALLEN. Second edition. Vol. III., Part III. London: J. and A. Churchill. Price 16s.

VOL. III., Part III. of Mr. Allen's *magnum opus* fully maintains the high-water mark of the preceding volumes; and perhaps no higher praise than this could be awarded to it. The Part III. now before us is indeed in reality Vol. V. of the greatest treatise on Commercial Organic Analysis that has ever appeared in this country. Why a work which is of the utmost value to every scientific analyst should be

dubbed with the term "Commercial" we hardly know; but we are sure that an analyst can venture on no better commercial speculation than to purchase this book, which for fulness of detail, accuracy, and scientific breadth of treatment leaves little to be desired.

In Part III. the analysis of vegetable alkaloids is concluded, the account of their reactions, etc., extending over 89 pages. As samples of the care with which Mr. Allen has dealt with this branch of the subject, we may refer our readers to the section dealing with the alkaloids of laburnum and furze, and to that on the alkaloids of ipecacuanha, as examples. These sections are thoroughly up to date in their treatment, and in them will be found the newest researches of Pantheil, Gerrard, and Paul and Cownley.

The Non-Basic Vegetable Bitter Principles are next dealt with, and all that is known with certainty about the chief of these somewhat obscure bodies will be found recorded. We may instance the articles on the glucosides of digitalis and of strophantus as admirable specimens of Mr. Allen's treatment of his subject. It seems a little odd, however, to meet with the adulterations of mustard under the head of the glucosides of mustard, for we opine that these glucosides are not the bodies which the analyst usually estimates when he has to report on the adulteration of the condiment in question. But with respect to such a good memoir on mustard generally, it is hypercriticism to carp at the place assigned to mustard adulteration in the book under review.

Animal Bases occupy 160 pages of the book, and of these some 30 pages are devoted to ptomaines. Either the heading or the arrangement of this section is perhaps not happy, since it is difficult to understand why such bodies as asparagine and glutamine are placed among animal bases, until we see that they are conveniently treated of as amides, together with glycocine and leucine. The apparent anomaly then disappears.

In treating of ptomaines Brieger's work is closely followed, and making every allowance for the non-confirmation of some portions of his work by others, Brieger's classical memoirs on the Ptomaines stand unequalled for completeness. Unhappily, the discredited work of some other experimenters finds more than its due recognition by Mr. Allen. The practical analytical points are, however, duly noted by the author, and especially the behaviour of ptomaines towards alkaline bicarbonates, and the precautions to be adopted in their separation. The toxicological analyst will find in this section invaluable help.

After a concise review of the animal acids, the volume under review concludes with a section on cyanogen and its derivatives, in which the author is very much at home. In one point only, from the "commercial" point of view, do we find this section defective. The author does not give us any warning of the not infrequent presence of sulphocyanides in sewage, and its detection and estimation therein. On several occasions where it was noticed that sewage, when applied to land, proved inimical to vegetation, we have found sulphocyanides—no doubt derived from gas liquors—in the harmful sewage.

Chemists will hail with gratitude the promised concluding volume of Mr. Allen's monumental work.

T. S.

THE ANALYST.

NOVEMBER, 1896.

NOTE ON THE CONCENTRATION OF CONDENSED MILK.

BY ALFRED H. ALLEN.

IN the case of unsweetened condensed milk the method of calculating the concentration which has been effected, and the water required to dilute the product to its original strength, is of the simplest possible character. But in the case of sweetened condensed milk certain factors exist which, if not taken into account, may lead to a serious miscalculation as to the concentration. Thus, the "Milkmaid" brand, which is a typical and well-known make of sweetened condensed milk, contains, according to the analysis of Pearmain and Moor, 37.6 per cent. of milk-solids, 38.7 per cent. of cane-sugar, and 23.7 per cent. of water. The milk-solids are three times as high as in uncondensed milk of good quality, which may be taken as containing 12.5 per cent. of solids and 87.5 per cent. of water. The water originally associated with 37.5 per cent. of milk-solids would be $37.5 \times 3 = 262.5$, whereas the condensed milk contains only 23.7 parts of water for the same amount of milk-solids. Therefore, 238.8 parts of water out of 262.5 originally present, or about ten-elevenths of the whole, must have been evaporated. But the addition of cane-sugar dilutes the milk thus concentrated, so as to produce a finished product containing only three times the proportion of milk-solids present in unconcentrated milk. Hence the addition of twice its weight of water to the condensed milk will reduce the milk-solids to their original proportion. But, as the dilution is in practice effected by measure rather than by weight, the number of measures of water (W) required to reduce one measure of condensed milk to its original concentration may be found by the following equation:

$$\text{Measures of water required for dilution (W)} = \frac{\text{Milk-solids in condensed milk (C)} \times \text{sp. gr. (G)}}{\text{Milk-solids in diluted milk (D)}} - 1.$$

Sweetened condensed milk of the character of the "Milkmaid" brand has a specific gravity of about 1.28. If this figure be accepted, and the milk-solids of the original milk be taken at 12.5 per cent., then the above equation becomes:

$$W = \frac{C \times 1.28}{12.5} - 1; \text{ or } W = 0.1024C - 1.$$

Taking as an example the "Milkmaid" brand with 37.6 per cent. of milk-solids, somewhat less than three measures of water will reduce it to the primary concentration:

$$W = (0.1024 \times 37.6) - 1 = 2.85.$$

Similarly, the percentage of milk-solids which will be contained in a diluted condensed milk may be found by the equation :

$$\text{Milk-solids in diluted milk (D)} = \frac{1.28C}{W+1}.$$

Thus, taking the "Milkmaid" brand, if the milk be diluted with the maximum quantity of water directed on the label, namely, 14 parts, the diluted milk will contain 3.21 per cent. of milk-solids :

$$\frac{1.28 \times 37.6}{14+1} = 3.21.$$

If for the value of C in the above equation the percentage of fat (F) in the condensed milk be substituted, the proportion of fat in the diluted milk will be found. In the case of the "Milkmaid" brand, diluted to the maximum as before, this will be :

$$\frac{1.28 \times 11.0}{14+1} = \frac{14.08}{15} = 0.94 \text{ per cent. of fat in the diluted milk.}$$

The foregoing equations give the dilution necessary to bring the parts of milk-solids per 100 measures of the diluted condensed milk to that contained in the original milk before concentration. If unity, when it appears in the above equations, be multiplied in each case by the specific gravity of the condensed milk (= 1.28), the equations will give the dilution necessary to yield a product containing 12.5 per cent. by weight of milk-solids.

The following table shows the proportion of fat which would be contained in representative brands of sweetened condensed milk if the contents of the tin were diluted with the amount of water directed on the labels. The calculations have been based on the assumption that the condensed milk before dilution had in each case a specific gravity of 1.28. This is the result of experiment on typical preparations of the kind.

Brand.	Number of parts of Water recommended to be added to one part of Condensed Milk for cooking and ordinary use.	Percentage by weight of Fat in Milk thus diluted.	Number of parts of Water recommended to be added to one part of Condensed Milk for infants' use.	Percentage by weight of Fat in Milk thus diluted.
A Alderney	5 to 6	2.32 to 1.94	6 to 8	1.94 to 1.52
B Arcadia	4 to 5	1.95 to 1.63	7 to 14	1.24 to 0.68
C Cowslip	4 to 5	0.34 to 0.28	Not stated	Not stated
D Devon	4 to 5	2.06 to 1.72	6 to 14	1.50 to 0.71
E Farm	4 to 5	0.03 to 0.02	Not stated	Not stated
F Fourpenny	4 to 5	2.52 to 2.10	7 to 14	1.60 to 0.87
G Full weight ...	4 to 5	2.81 to 2.34	7 to 14	1.79 to 0.97
H Goat ...	5	0.87	Not stated	Not stated
I Milkmaid	4 to 5	2.66 to 2.22	7 to 14	1.69 to 0.92
J Nestle's Swiss	4 to 5	3.30 to 2.77	Not stated	Not stated
K Rose ...	4 to 5	3.00 to 2.50	7 to 14	1.91 to 1.04
L Threepenny...	4 to 5	0.07 to 0.06	Not stated	Not stated

THE RELATIVE COMPOSITION OF MILK, CREAM, AND SKIMMED MILK.

BY NORMAN LEONARD, B.Sc., F.I.C., AND HARRY M. SMITH, F.I.C.

As a layer of cream gradually forms on the surface of milk, the composition of the liquid varies at different levels. The upper layers contain a larger proportion of fat, and a smaller proportion of other constituents, than the original milk, while in the lower layers these conditions are reversed. If the change which takes place during the rising of the cream consist solely in the ascent of fat globules, it is clear that the relation between the amounts of water and of solids-not-fat will be the same in the cream and in the skimmed milk as it was in the original whole milk. The lower layers, in fact, may be regarded as an aqueous solution of milk-sugar, casein, and other substances, containing little fat in suspension, while the upper layers consist of a precisely similar solution, with a larger amount of suspended fat.

Wanklyn, in his book on milk analysis, adopts this view, and gives a few perhaps not very conclusive figures in support of it. He states, however, that rich town-fed milk is apt to throw up a little casein with the cream, and points out at the same time that any imperfection in the analysis of cream tends to raise the solids-not-fat. H. Droop Richmond, in giving results of analyses of cream, has also referred to this point on various occasions. In the rich so-called "Devonshire" creams there seems to be little doubt, judging from the many recorded analyses, that there is a larger amount of solids-not-fat, in proportion to the water, than is to be found in ordinary whole milk. The heat commonly applied to assist the separation of these rich creams may possibly be the cause of this "incipient coagulation."

The question is obviously of some importance to those connected with the working of the Sale of Food and Drugs Act; for if casein be thrown up with the cream, a sample of milk taken from a vessel, the contents of which have been imperfectly mixed, cannot by analysis afford any trustworthy evidence either as to the addition of water or deficiency in fat. If, however, the relation between the water and the solids-not-fat be undisturbed, the analysis will show the presence or absence of added water, although, of course, no conclusion can be drawn as to the fat. It was a discrepancy between the analyses of samples stated to have been drawn from the same churn at different times which induced us to make some experiments with a view to determining whether Wanklyn's theory held good or not. That it does hold, under ordinary conditions and so far as the usual analytical methods are capable of deciding, is, we think, proved by our results, which are given in the following table:

						Calculated on Water.	
						Solids-not-Fat.	Ash.
			Total Solids.	Fat.	Solids-not-Fat.	Ash.	Water.
A						10.28	0.84
A 1	$\frac{1}{3}$...	12.66	3.68	8.98	0.75	87.34
A 2	$\frac{2}{3}$...	15.95	7.37	8.58	0.71	84.05
A 3	$\frac{2}{3}$...	11.05	1.91	9.14	0.78	88.95
A 4	$\frac{1}{3}$...	13.67	4.83	8.84	0.74	86.33
A 4	$\frac{1}{3}$...	10.63	1.58	9.05	0.76	89.37
B						7.22	0.60
B 1	$\frac{1}{3}$...	9.60	3.07	6.53	0.54	90.40
B 2	$\frac{1}{3}$...	16.44	10.16	6.28	0.45	83.56
B 3	$\frac{1}{3}$...	8.32	1.68	6.64	0.54	91.68
B 3	$\frac{1}{3}$...	7.67	0.91	6.76	0.55	92.33
C						9.75	0.79
C 1	$\frac{1}{7}$...	11.60	2.98	8.62	0.70	88.40
C 2	$\frac{6}{7}$...	20.10	12.39	7.71	0.64	79.90
C 2	$\frac{6}{7}$...	9.28	0.60	8.68	0.78	90.72
						9.57	0.86

The samples of milk were allowed to stand in separators for about eighteen hours at the temperature of the laboratory. Measured portions were then drawn off successively from the bottom and submitted to analysis. In the first experiment a milk yielding on analysis the results given at A (see Table) was thus divided into two portions, an upper portion (A 1) forming one-third, and a lower portion (A 2) forming two-thirds of the original volume. In a second experiment, made with the same milk, the upper and lower portions (A 3 and A 4) formed respectively two-thirds and one-third of the whole. Another sample of milk (B) in the third experiment was divided into three portions, upper, middle, and lower, each of which was analysed; while in the last experiment on a milk (C) only the upper and lower portions, each forming one-seventh of the whole, were examined.

The numbers in the seventh and eighth columns of the table are obtained by dividing the percentages of solids-not-fat and of ash respectively by the percentage of water in the various samples, and multiplying the quotients by 100. The numbers in the seventh column are practically identical in each of the three sets of analyses. What variations there are exhibit no regularity, and are such as might be expected to be due to experimental error. The figures in the eighth column show a less satisfactory agreement. In each case the skimmed milk yields a higher proportion of ash than the cream. This is probably due to the subsidence of suspended mineral impurities (dust) in the milk.

The conclusion we draw from our experiments is, that at the ordinary temperature no disturbance of the relation between water and solids-not-fat takes place when milk is allowed to stand at rest, even when the upper layer contains twenty times as much fat as the lower. We would further suggest that, in estimating the amount of added water (if any) present in samples of milk containing an abnormally high or low proportion of fat, it is desirable to take as a basis for the calculation the percentage of solids-not-fat in the *water* instead of in the *milk*. The percentage of

solids-not-fat contained in the water of milk of average quality is, of course, taken as a standard for comparison.

We have to express our thanks to Dr. Stevenson, in whose laboratory these experiments were carried out.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Potassium Chromate as a Milk Preservative. J. Froidevaux. (*Jour. Pharm. Chim.*, 1896, 155-158.)—The author describes experiments made to determine what amount of neutral potassium chromate is necessary for preserving milk for an appreciable time, and finds that at least 0.2 gramme per litre is required, an amount which gives to the milk an intense and absolutely abnormal colour. He considers that the proportion of 2 grammes to 50 litres of milk, which (according to Denigès) the retailers of Bordeaux employ, is altogether insufficient to retard coagulation.

With regard to the detection of chromates in milk, the method of Denigès (addition of 1 c.c. of a 2 per cent. solution of silver nitrate to 1 c.c. of milk) is satisfactory when the amount of chromate exceeds 0.01 gramme per litre; when below this the colour is masked by the precipitated phosphates. The following method is preferred: The ash from 10 c.c. of milk is dissolved in a few drops of water acidified with nitric acid, neutralized with magnesium carbonate, and the silver nitrate solution (preferably 20 per cent.) added.

As a control test the ash from 10 c.c. of milk is taken up in a few drops of water slightly acidulated with sulphuric acid, and tincture of guaiacum added little by little. An intense blue colour, which rapidly disappears, is produced when chromates are present. The reaction will detect 0.02 to 0.03 gramme of chromate per litre.

C. A. M.

Detection of Formaldehyde in Milk. C. Denigès. (*Bull. Soc. Pharm. de Bordeaux*, July, 1896, p. 212; through *Ann. de Chimie Analyt.*, i. [16], 316.)—The colour reaction (red) given by bleached fuchsine with formaldehyde may be distinguished from that due to casein and albuminoids by changing into blue on the addition of hydrochloric acid, whereas the acid destroys the colour in the other case.

To perform the test 1 c.c. of fuchsine solution decolorized by sulphurous acid is added to 10 or 12 c.c. of milk. After 5 or 6 minutes 2 c.c. of pure hydrochloric acid are added, and the mixture shaken up. The violet-blue coloration indicating formaldehyde will be appreciable, even when only 2 or 3 centigrammes are present per litre. In the absence of this body, the mixture will become yellowish-white. The delicacy of the test may be heightened by diluting the milk with an equal volume of water, adding 3 to 4 drops of glacial acetic acid, followed by 5 c.c. of mercuric iodide solution; then agitating and filtering, and applying the fuchsine test to the clear filtrate, the acid being added after an interval of 10 minutes. In this way a

visible coloration will be obtained in the case of 1 centigramme of formaldehyde per litre of milk, especially if the liquid be examined by looking down the tube. The colour may be compared with standards corresponding to known percentages of formaldehyde.

The reagent is prepared from 40 c.c. of a $\frac{1}{2}$ per cent solution of fuchsine, 250 c.c. of distilled water, 10 c.c. of 40° B. sodium bi-sulphite and 10 c.c. of pure sulphuric acid. The mixture will be sufficiently decolorized for use after standing a few hours.

C. S.

Detection and Estimation of Sodium Bicarbonate in Milk. L. Pade. (*Ann. de Chimie Analyt.*, i., [17], 328.)—The best method of detecting the presence of added sodium bicarbonate in milk is by determining the alkalinity of the soluble ash, but this method is insufficient for a quantitative determination, since the greater portion of the sodium enters into combination with phosphoric acid during the process of incineration, necessitating the titration of the phosphoric acid also.

The ash from 25 c.c. of milk is dissolved in water, and the alkalinity of the solution determined by $\frac{1}{10}$ normal sulphuric acid, the volume of which, multiplied by 0.0084 gives the quantity of unaltered sodium bicarbonate in the 25 c.c.; or the percentage may be obtained direct by multiplying by 0.0336. After adding to this neutral solution about 2 c.c. of a 10 per cent. solution of sodium acetate slightly acidified with acetic acid, the phosphoric acid is titrated by uranium acetate solution, standardized to correspond bulk for bulk with a solution containing 3.11 grammes of sodium-ammonium phosphate per litre, so that 1 c.c. of the uranium solution is equivalent to 0.01 gramme of sodium bicarbonate per 100 c.c. of milk. Cochineal tincture forms the most delicate indicator. Proceeding in this manner, the author has obtained results not exceeding by more than 0.008 gramme per 100 c.c. of milk the quantity of bicarbonate added.

C. S.

The Detection of Borax in Butter. Planchon and Vuafart. (*Jour. Pharm. Chim.*, 1896, 49-51.)—Twenty grammes of the butter are melted in a basin, dissolved in 10 c.c., of petroleum spirit, and the solution poured into a separating funnel. The basin is rinsed out into the funnel first with 10 c.c. of petroleum spirit, and then with 10 c.c. of water. After being well shaken the liquids are allowed to separate, and the aqueous layer, together with the supernatant film of casein, run into a platinum basin, evaporated to dryness and incinerated. The ash is fused with 0.5 gramme of pure dry potassium carbonate, a very minute quantity of copper oxide added, and the fusion repeated. On cooling, a blue colour, more or less pronounced, denotes the presence of borax. The shade varies from greenish to violet-blue. Pure butter treated in this way gives only a gray or reddish-gray colour. Phosphates and fluorides do not give the coloration, but silicates dissolve the copper oxide and yield a pale blue mass which, however, is quite distinct from that produced by borax. As regards the sensibility of the reaction, a distinct colour is invariably given with two parts of borax per 1,000, and sometimes with 1 per 1,000, while by taking a larger quantity of butter still smaller proportions may be detected.

The method has not proved very satisfactory in the case of milk, wine and beer. With milk the difficulty of fusing the ash makes it necessary to increase the amount of potassium carbonate, and the fused mass is then too voluminous to show the colour plainly. The difficulty with wine and beer is that the ash alone when fused with potassium carbonate produces a green colour, which is probably due to a trace of manganese.

C. A. M.

Examination of Butter. C. Aschman. (*Chem. Zeit.*, 1896, xx., 723.)—By the following process margarine may readily be distinguished from genuine butter. 5 grammes of the sample are saponified in a basin in 10 c.c. of 95 per cent. alcohol and 2 c.c. of 50 per cent. potash, and the soap is dissolved in about 150 c.c. of water. The solution is poured into a 300 c.c. flask provided with a mark at 200 c.c., 4 c.c. of 1 : 3 sulphuric acid run in, and when cold made up to the lower mark; 60 c.c. of ether are added, the whole well shaken two or three times, the flask closed with a cork, and stood in water at 15° C. 30 c.c. of a 30 per cent. solution of common salt (sp. gr., 1.175) are put into a stoppered tube about 40 cm. long, holding 100 c.c., 8 c.c. of decinormal potash, and 20 c.c. of the above ethereal solution added, and after a thorough agitation the mixture is set aside for one or two hours till separation has taken place. On examination the ether will be found to contain a layer of matter insoluble in both liquids which, with genuine butter, will not be more than 20 or 25 mm. thick; but in the case of margarine will be 60 or 70 mm., if indeed the whole of the ether be not filled with it.

F. H. L.

Contributions to the Analysis of Honey. E. Beckmann. (*Zeit. anal. Chem.*, 1896, xxxv., 263-284.)—After a short summary of work done by others on this subject, the author describes experiments made with the object of detecting "starch syrup" and commercial dextrin in honey.

Detection by means of Methyl Alcohol and Iodine Solution.—On treating diluted "starch syrup" with methyl alcohol (or acetone) a white precipitate was obtained which could be dried by repeated evaporation on the water-bath with ethyl alcohol. The dextrin (about 31 per cent.) was a white non-hygroscopic powder with a slightly sweet taste. When tested with a solution of iodine in potassium iodide it gave the reddish-brown colour of erythro-dextrin. In the manufacture of the syrup the inversion of the starch by the acid is not carried beyond the point where iodine gives the red reaction. Honey treated with the above reagents only gave a light flocculent turbidity which did not settle on the walls of the vessel like the precipitate from "starch syrup." A mixture of honey and dextrin gave a large precipitate, and this (as well as the original honey) gave an intense red or violet colour with iodine solution.

Dialysis of "Starch Syrup" and Honey.—Hänle's method (*ANALYST*, xvi., 79) was employed to see whether the substances precipitated by methyl alcohol were also diffusible. It was found that throughout the operation portions of the dextrinous bodies passed through the membrane. The author considered that no sharp separation of honey-dextrins and starch-dextrins was possible by the method, although the

time taken by honey to completely diffuse was considerably shorter than that required by the starch products. When dialysed with water at a higher temperature, 50-70° C., the "starch syrup" diffused more slowly than the honey, but in this case also the whole of the constituents eventually passed through the membrane. Dialysis with warm methyl alcohol gave the following results :

	Honey 10 grammes.	5 grammes Honey + 5 grammes Starch Syrup.	10 grammes Syrup.
Residue :	After 50 hours	After 103 hours	After 103 hours
	0.0 gramme.	2.2 grammes.	4.8 grammes.

Consecutive treatment with methyl and ethyl alcohols.—(1) *Starch syrup.* After filtering off the methyl alcohol precipitate, the addition of ethyl alcohol to the filtrate produced a fresh precipitate, differing from the former one in being hygroscopic—80 grammes of syrup dissolved in 80 c.c. of water gave, with methyl alcohol, a precipitate of 25 grammes = 31 per cent. The filtrate, after evaporating the methyl alcohol and adding ethyl alcohol, gave 33 grammes = 41 per cent. (2) *Conifer honey*, $[\alpha]_D = +16.9^\circ$. When treated with ethyl alcohol as above yielded 50 per cent. of a strongly hygroscopic substance. Conifer honey was practically soluble in methyl alcohol. (3) *Solid glucose.* In this the starch had been inverted until complete disappearance of the red colour with iodine. Methyl alcohol produced only a slight turbidity, but ethyl alcohol precipitated 46.3 per cent. of a very hygroscopic dextrinous substance. (? Gallisin. Iso-maltose.)

Fermentability of the different dextrins.—Although the dextrins of honey were more fermentable than those of "starch syrup" or glucose, the author considered that no certain conclusion could be based on this alone. None of the residues left after fermentation gave a red colour with iodine—a proof that the erythro-dextrin was further hydrolyzed.

Precipitation with methyl alcohol and baryta.—The dextrins of honey behaved differently to those of "starch syrup," etc., in the formation of dextrinates, and on this property the following test was based: 5 c.c. of honey solution (20 grammes in 100 c.c. water) were mixed with 3 c.c. of a 2 per cent. solution of barium hydroxide, and 17 c.c. of methyl alcohol added. On shaking, the solution remained clear, or only slightly turbid when pure honey was present, but with starch-dextrin, "starch syrup" or glucose, there was a considerable precipitation. For a quantitative estimation the amount of honey taken was increased to 50 grammes, the methyl alcohol added rapidly all at once to avoid deposition on the glass, the liquid well shaken *once*, and the precipitate filtered on to a tared asbestos filter, washed with methyl alcohol and ether, and dried at 55° to 60° C. Excessive shaking was avoided to prevent the action of air on the precipitate, and it was found that the quicker the working the more accurate the results. Only in exceptional cases did the presence of sulphates or phosphates in the honey interfere with the results, and these were then separately determined and allowed for.

The mean results obtained in this way, calculated on 1 gramme of the substance, were: Dextrin, 0.916 gramme; "starch syrup," 0.455 gramme; glucose, 0.158 gramme. The amount of precipitate produced was in each case nearly proportional to the concentration of the solution. The admixture of conifer honey with the above

three substances in the proportion of 90, 75, and 50 per cent. did not increase the amount of the precipitates, but, on the contrary, somewhat diminished them.

Various pure samples of natural honey, rich in dextrin, were tested in this way :

Apple Honey.— $[\alpha]_D = -12.2^\circ$. Dextrin precipitated by alcohol (König and Karsch) = 23.7 per cent.

Baryta precipitate : 5 c.c. of 10 per cent. solution gave 0.0044 gramme.

5 " 20 " " 0.0072 "

Umbellifer Honey.—Dark, with green fluorescence. $[\alpha]_D = -4.610^\circ$. Dextrin = 29.1 per cent.

Baryta precipitate : 5 c.c. of 10 per cent. solution gave 0.0148 gramme.

5 " 20 " " 0.0230 "

Conifer Honey.—Slightly resinous smell. $[\alpha]_D = +16.9^\circ$. Dextrin = 41.5 per cent.

Baryta precipitate : 5 c.c. of 10 per cent. solution gave 0.0132 gramme.

5 " 20 " " 0.0248 "

Hence the author concluded that under the most unfavourable conditions one could recognise the addition to conifer honey containing over 40 per cent. of dextrin of from 5 to 10 per cent. of starch dextrin, from 10 to 20 per cent. of "starch syrup," and of 30 to 40 per cent. of glucose. With ordinary honey, like the above apple honey, adulteration would be much more easily detected.

By a combination of the fermentation and precipitation methods very small addition of starch products would be detected in doubtful cases.

Honey with Honey-dew.—On treatment with iodine solution honey-dew gave but a slight colour, but otherwise behaved like "starch syrup." Methyl-alcohol precipitated dextrans which deposited on the glass, and large barium precipitates were obtained. (Cf. ANALYST, xx., 16.)

Honey from Bees fed on "Starch Syrup."—The honey thus obtained behaved in a similar way to normal honey, showing that the bees can so modify starch dextrans that they assume the character of honey dextrans.

Detection of Cane-sugar and Molasses.—Bees cannot completely invert cane-sugar, which is therefore a normal constituent of honey. What amount is allowable is uncertain, since Utescher gives 5 per cent. as the maximum, whilst Hefelmann and the Swiss chemists allow as much as 16 per cent.

For detecting molasses the author used basic lead acetate and methyl-alcohol, which gave white precipitates attributed to the presence of raffinose. It was found that with honey the concentration must not exceed 25 per cent.

In this way the addition of 10 per cent. of molasses to honey could be easily detected.

C. A. M.

Detection of Foreign Colouring Matters in Red Wines. A. Belar. (*Zeit. anal. Chem.*, 1896, xxxv., 322, 323.)—Owing to the fact that many of the aniline colours are soluble in nitro-benzene, whilst the blue and red colouring matters of plants are quite insoluble, the former can often be detected in red wines. About 5 c.c. of the wine are gently shaken with an equal volume of nitro-benzene; when in the presence of fuchsin the nitro-benzene assumes a red colour. Should it remain colourless, the shaking is repeated more vigorously, and the emulsion got rid of by

gently warming. By using a sufficient quantity of nitro-benzene fuchsin may be completely extracted from a wine and quantitatively estimated.

Methylene blue is extracted from an aqueous solution by nitro-benzene, to which it imparts an emerald green tint, while rosanilin, purpurin and safranin dissolve without changing their colour. Eosin is largely soluble, giving a wine-red colour, but the solution does not fluoresce. The insoluble portion has a yellow tint, as has also the portion of rosolic acid not extracted by nitro-benzene.

Indigo-carmin, on the other hand, behaves like the blue colouring matters of plants, being quite insoluble in nitro-benzene.

C. A. M.

The Amount of Copper absorbed by Vegetables from a Coppery Soil. K. B. Lehmann. (*Arch. Hygiene*, 1896, xxvii., 1; through *Chem. Zeit. Rep.*, 1896, 245.)—The author still maintains that Vedrödi's results (*ANALYST*, xxi., 235) are too high. He now burns his samples with or without the addition of sulphuric acid, dissolves the ash in nitric acid—fusing any residue with soda and saltpetre, dissolving in acid, and mixing the two solutions—precipitates with sulphuretted hydrogen, ignites the sulphide, dissolves in hydrochloric acid, and determines the copper colorimetrically as before. He finds that the amount of copper in the vegetables diminishes rapidly with the distance from the source of contamination, and that the species of plant has far less influence than the quantity of copper in the soil on the amount taken up. In woody plants the bark contains most metal, and the wood least. Half of the copper is soluble in water, but it exists as some organic compound, perhaps with albumin, which prevents its being detected in the solution by ordinary reagents. The metal appears neither to promote nor to hinder the growth of the plants.

F. H. L.

Rapid Estimation of Zinc in Articles of Food. Janke. (*Chem. Zeit.*, 1896, xx., 800.)—Fifty to 100 grammes of the substance (*e.g.*, apple rings) are cut up into pieces, dried for three hours at 125° C., and then powdered. 25 c.c. of 1.31 nitric acid and 10 c.c. of strong sulphuric acid are added, and the whole is ignited for about four hours in a covered platinum crucible at a barely visible red heat. The resulting white ash is moistened with nitric acid, evaporated on the water-bath, taken up in water, filtered, the iron, etc., removed by the ammonium acetate process, and finally the zinc in the filtrate is precipitated twice as sulphide. Twelve analyses of samples containing 10 to 50 milligrammes of ZnO are quoted, the amount recovered varying from 98.8 to 104.5 (mean 100.41) per cent. (*Cf. ANALYST*, xx., 251.)

F. H. L.

Adulteration of Cantharides. M. Cabannes. (*Bull. Pharm. du Sud-Est*; through *Jour. Pharm. Chim.*, 1896, 310, 311.)—It is stated that cantharides are frequently adulterated with insects of similar appearance. One sample was found by the author to consist of four varieties: *Cantharis vesicatoria* (25 per cent.); *C. togata* (45); *Sylpha quartapunctata* (20); and *Cetonia aurata* (10). Of these the two last were completely devoid of vesicating properties, whilst *C. togata* only con-

tained 0.27 per cent. of cantharidin, instead of 0.50 per cent., as in the genuine beetle. This variety, which has its origin in Turkestan, is larger than *C. vesicatoria*; its abdomen is more tapering, and it has on the elytra a longitudinal yellow band rarely reaching the extremity. When applied to the skin it only produces a slight redness after the lapse of twenty-four hours. The *C. vesicatoria* of the above-mentioned mixture did not contain the amount of cantharidin required by the *Codex*, having only 0.4115 per cent. instead of 0.50 per cent. C. A. M.

The Detection of Acetanilide. F. X. Moerk. (*Amer. Journ. Pharm.*, 1896, lxxviii., 389-393.)—To obtain a method for the detection of acetanilide in closely allied synthetic remedies the author studied the action of bromine on 1 per cent. solutions of the various compounds. When bromine-water was added drop by drop so long as the colour was discharged, and the liquid stirred, after five minutes the results were :

	C.C. Bromine- Water added.	Appearance.
Acetanilide	2.0	{ Yellow liquid, white precipitate.
Exalgin	0.8	{ Colourless liquid, white precipitate.
Methacetin, $C_6H_4(OCH_3)NH(COCH_3)$	1.8	{ Yellow liquid, becoming
Phenacetin	0.6	{ colourless, pink, finally
Phenocoll	2.0	{ red-brown, slightly turbid.
Lactophenin, $C_6H_4(OC_2H_5)NH(COCHOHCH_3)$	1.2	{ Liquid like above; distinct white precipitate.
Salophen, $C_6H_4(C_7H_5O_3)NH(COCH_3)$	{ Colourless, slightly turbid liquid.

On shaking with a light petroleum benzine, the exalgin precipitate readily dissolved, the acetanilide precipitate slightly dissolved, whilst the lactophenin precipitate was insoluble. Ether was not so satisfactory, as it dissolved all the precipitates. Since a precipitate could be obtained with a solution containing 1 in 2,000, the bromine test seemed to be applicable to the detection of not less than 5 per cent. of acetanilide in all the remedies except lactophenin.

The isonitrile test was modified so as to destroy the odours due to other remedies. 0.1 gramme of methacetin, phenacetin, lactophenin, salophen, or phenocoll hydrochlorate, was boiled with 10 c.c. of water (salophen being the only one insoluble), the liquid cooled quickly and filtered through cotton. To 2-3 c.c. of the filtrate an equal volume of a 5 per cent. solution of potassium or sodium hydrate was added, with some crystals of potassium permanganate, until a violet colour remained after boiling. Two or three drops of a mixture of chloroform 10 c.c., alcohol 10 c.c., and ammonia solution 0.5 c.c. were then added and the liquid boiled, more of the chloroform mixture being added when the permanganate had not been completely reduced. After standing for a few moments the odour was noted and in doubtful cases compared with that yielded by a dilute acetanilide solution.

In testing exalgin the potassium permanganate was omitted. By this test the author found that 1 per cent. of acetanilide could be readily detected in these allied compounds (*cf.* Hyde, *ANALYST*, xxi., 69). C. A. M.

The Alkaloids of the Kola-nut. Carles. (*Ann. de Chimie Analyt.*, i. [18], 345-347.)—The accuracy of the estimation of caffeine and theobromine in Kola-nuts depends much on the state of division and dryness of the powdered nut, as well as on the solvent employed for the extraction. It is essential that the nut should be powdered so fine as to pass through a No. 0 (120 mesh) silk sieve, and that it should contain about 25 to 30 per cent. of moisture, otherwise only part of the caffeine will be extracted by chloroform. In view of the sluggish action of this solvent on the alkaloids of the Kola-nut, it is preferable to add to it 20 per cent. of 93-94 per cent. alcohol, which facilitates solution and preserves the requisite degree of humidity. An admixture of lime assists in the liberation of the alkaloids which exist in combination with kolatannic acid as kolanine, and at the same time, by destroying the horny texture of the nut, facilitates lixiviation.

In place of a Soxhlet apparatus, an ordinary 100 c.c. flask may be used for the extraction, a straight or bent tube of 1 metre in length sufficing as condenser.

Ten grammes of the sifted Kola-nut powder are mixed thoroughly with 1 gramme of calcium hydroxide and 20 grammes of 80 per cent. alcohol. After drying over the water-bath, until the weight of the mixture is reduced to 14 grammes, the powder is re-ground and heated in the 100 c.c. flask for an hour over the water-bath, with 35 c.c. of the alcohol-chloroform mixture, the solution and residue being separated by filtration, and the latter re-extracted three times in the same way, with respectively 35, 30, and 20 c.c. of the solvent mixture. The united extracts are evaporated to dryness, taken up with 10 c.c. of boiling water (containing 4-6 drops of 1 per cent. sulphuric acid), then by another 6 c.c., and finally by 5 c.c., of boiling water, filtered and dried at 100° C. until constant. The weight represents the caffeine plus theobromine in the 10 grammes of powder taken.

To determine the kolanine in the nut or nut extract, the caffeine may be extracted by means of cold water and the kolanine dissolved by 70 per cent. alcohol. On evaporating the alcoholic solution down to the consistency of an extract, and then taking this up with cold water, the kolanine will be left behind, and may be dried at a gentle heat until its weight is constant. The amount of alkaloids contained in the kolanine may be estimated by triturating 1 gramme with an equal weight of calcium hydroxide and a little 70 per cent. alcohol, adding 3 grammes of chalk to increase the division of the substance, and, after drying down to 6 grammes, proceeding with the extraction by alcohol and chloroform.

C. S.

Estimation of Phosphoric Acid in Medicated Wines. F. Glaser and K. Mühle. (*Chem. Zeit.*, 1896, xx., 723.)—In working the processes that Fresenius has described for this purpose, whether the sugar contained in the wine has been removed by fermentation or not, it is not sufficient that the dried residue be carbonized, it must be ignited till it is perfectly white. Repeated extraction of the carbonaceous matter with strong nitric acid only recovers a portion of the phosphoric acid, which would appear to be present in some insoluble modification. Although these methods are very accurate, provided the directions are followed exactly, yet they are tedious and require great care, while the author's process is simpler and gives equally satisfactory results.

100 c.c. of the wine are evaporated to a syrup in a 250 c.c. flask of Jena glass. After cooling, 25 c.c. of strong nitric acid are added, and the liquid is cautiously warmed till decomposition begins. When the evolution of gas ceases, 75 c.c. more acid are introduced, and the whole is evaporated over a naked flame until it is almost dry. The vessel is then cooled, 10 c.c. of strong sulphuric acid and a drop of mercury added, and the liquid is heated gently till it darkens in colour and the organic matter is destroyed. The mixture is again cooled, diluted to the mark, filtered, and after 100 c.c. of the filtrate (40 c.c. of wine) have been neutralized with ammonia, the phosphoric acid is thrown down with either molybdate or magnesia mixture. Up to the time of precipitation the process takes only three hours.

F. H. L.

TOXICOLOGICAL ANALYSIS.

On the Identification of Morphine in Toxicological Cases. J. B. Nagelvoort. (*Amer. Jour. Pharm.*, 1896, lxviii., 374-379.)—In order to determine how small a quantity of morphine could be isolated from putrefying animal matter, test mixtures were prepared. In one experiment $\frac{1}{2}$ grain of the alkaloid was dissolved in a plate of soup. In others 0.050 gramme of morphine was mixed with refuse from a restaurant and with human flesh. These were left to putrefy for fifty days in a warm room, and at the end of that time examined by the methods of Dragendorff, Stas-Otto, and Graham (dialysis). Ptomaines were removed by washing the aqueous acid and aqueous alkaline liquids with ethyl ether and with a mixture of 4 volumes of ethyl ether and 1 volume of chloroform. Isobutylic alcohol was substituted for amyl alcohol to avoid the unpleasant effects of the latter. One mixture was examined by Kippenberger's glycerin tannin method (*ANALYST*, xx., 201). In every instance the morphine was separated and identified by satisfactory tests.

The conclusion arrived at is that the popular belief in the destruction of alkaloids during cadaveric putrefaction, so far as morphine is concerned, has no foundation in fact, and in this the author confirms the statements of Davoll (*ANALYST*, xx., 38) and of Kippenberger (*ibid.*, xx., 201).

C. A. M.

ORGANIC ANALYSIS.

Alcohol as a Source of Error in the Titration of Alkaloids. C. Caspari, Jr. (*Amer. Jour. Pharm.*, 1896, lxviii., 473-481.)—Having observed during some analytical work that alcohol appeared to influence the colour produced by acids and alkalies with different indicators in the titration of alkaloidal residues, the author made experiments to determine whether alcohol really was the cause of the variation. He found that with the indicators tried—hæmatoxylin, cochineal, Brazil wood, methyl orange, lacmoid, and litmus—dilute alcohol and absolute alcohol as available in the market exerted a decided influence. With methyl orange it seemed to play the part of an alkali, a large amount of decinormal acid being required to produce

the acid colour, whilst to the other indicators mentioned it gave a strong acid reaction.

The effect of alcohol on the results obtained in titrating alkaloids was proved by experiments with morphine, cocaine, atropine, and strychnine. Solutions were prepared containing 0.5 gramme of alkaloid and 20 c.c. of decinormal acid in 100 c.c., and 10 c.c. of the solutions used for each titration, the excess of acid being titrated with centinormal alkali. The percentages obtained were :

Indicator.	MORPHINE.		COCAINE.		ATROPINE.		STRYCHNINE	
	Water Solution.	Dilute Alcohol Solution.	Water Solution.	Dilute Alcohol Solution.	Water Solution.	Dilute Alcohol Solution.	Water Solution.	Dilute Alcohol Solution.
Hæmatoxylin ...	98.58	96.05	97.26	94.65	99.89	96.82	97.03	94.59
Cochineal ...	98.48	95.26	96.35	95.02	100.08	97.33	97.43	94.25
Brazil Wood ...	98.32	89.68	95.95	90.71	99.75	94.62	96.53	89.11
Methyl Orange ...	98.55	105.44	97.26	104.23	100.02	106.58	97.19	103.54
Lacmoid ...	98.91	97.56	97.44	96.53	100.38	97.95	98.03	97.19
Litmus ...	98.41	94.05	96.35	92.83	98.20	91.49	92.11	84.03

In the case of methyl orange the dilute alcohol solution required the addition of 1.5 c.c. of $\frac{N}{10}$ H_2SO_4 before a satisfactory end reaction could be obtained. The addition of 5 c.c. of alcohol to 10 c.c. of the dilute alcohol solution increased the errors still further.

Owing to the fact that quinine and cinchonine give an alkaline reaction with the indicators in acid solution, they cannot be estimated volumetrically in the same way as the other alkaloids, the results being altogether too high. Still, it was possible to prove that with these also the presence of alcohol influenced the result. A solution of quinine which, titrated in water, showed 117.18 per cent., gave 112.79 per cent. when titrated in a mixture of alcohol and water.

The author concludes that while it is possible to accurately titrate alkaloids and alkaloidal residues in alcoholic solution, it is necessary to know the exact amount of alcohol present, and the relation of the centinormal alkali to the decinormal acid, for the particular indicator used, in the presence of that amount of alcohol. It is therefore advisable to make the titrations in aqueous solution.

It is suggested that this behaviour of alcohol may be explained on the basis of Arrhenius's theory of electrolytic dissociation. According to Ostwald, indicators depend for their value entirely upon dissociation, and although alcohols have a dissociating effect upon salts held in solution by them, it is less marked than in the case of water, and decreases with the increasing molecular weight of the alcohol.

C. A. M.

Estimation of Ethers in Alcohols. Barbet and Jandrier. (*Rev. de Chimie Analyt.*, i. [19], 367.)—The aldehydes always present in industrial alcohols are resinified by the bases employed for the saponification of the ethers, and falsify the

titration of the latter by neutralizing a certain amount of alkali. The extent of this error varies with the aldehyde present, and also with the initial degree of alkalinity employed in the test. For example, when the $\frac{1}{10}$ normal potash amounts to 10 per cent. of the volume of alcohol under examination, it is found that one part of acetic aldehyde is equivalent to 0.18 of acetic ether, the coefficient for paraldehyde being 0.03, and for acrylic aldehyde 0.39; whereas when the ratio of potash is increased to 30 per cent. the coefficient of the last-named aldehyde becomes 0.70.

In view of the unreliability of these coefficients the authors sought to discover a reagent for saponifying the ethers, which should be without action on the aldehydes, and this they have found in calcium saccharate. A strong solution of this is prepared, employing 5 parts sugar to 1 of lime, and the mixture allowed to stand for 24 hours. It is then filtered and made up with sugar solution to decinormal strength.

To apply the test the alcohol is reduced to 50 per cent. with water in order to prevent in some measure the precipitation of the reagent. For industrial alcohols 10 per cent. by volume of the saccharate solution is employed. Saponification is effected by heating for two hours, after which the residual alkalinity of the liquid is titrated. The absence of resinification and coloration renders the end reaction distinct.

C. S.

Differentiation of Aldehydes by means of Phenols, and vice versa. Barbet and Jandrior. (*Ann. de Chimie Analyt.*, i. [17], 325.)—The phenols, in presence of chemically pure sulphuric acid, give characteristic and extremely delicate colour reactions with aldehydes. (*Cf. ANALYST*, xxi., 94.) To apply the test a few centigrammes of (*e.g.*) phenol crystals are placed in a tube and covered by about 2 c.c. of strong alcohol containing a trace of aldehyde. 1 c.c. of pure sulphuric acid is poured in gently down the side of the tube, and the resulting coloration at the plane of contact, and the change ensuing when the mixture is shaken up, are noted.

In the case of a 0.001 per cent. solution of acrylic aldehyde a yellow tinge forms in the acid layer, and a violet in the supernatant alcohol. When shaken up the yellow disappears, leaving a fine heliotrope shade.

Formic aldehyde may be distinguished from acrylic aldehyde by the negative results obtained with phenol, and by giving with gallic acid (which does not colour acrylic aldehyde) a yellow colour in the lower layer, rapidly changing to green by intermixture with the fine blue shade formed in the upper layer. When a 0.001 per cent. solution is in question the colour changes, on agitation, to heliotrope, passing into salmon red; with a 0.01 per cent. solution an intense dirty green coloration appears at the end of a few minutes.

The subjoined table exhibits the colorations obtained by such of the phenol reagents as have so far been found to give definite results. β -naphthol is extremely delicate as a group reagent, and indicates the presence of as little as 0.00005 per cent. of acetic aldehyde; it also gives a characteristic red coloration with benzoic aldehyde. Hydroquinone and phloroglucinol also serve to detect the aldehydes *en masse*, the former giving orange-yellow and the latter yellow colorations. For quantitative estimations 0.1 gramme of hydroquinone, 2 c.c. of the alcohol, containing the

Reagent.	Acetic Aldehyde.			Acrylic Aldehyde.			Pyromucic Aldehyde.			Commercial Formic Aldehyde.	Valeric Aldehyde.	Benzole Aldehyde.	Acetal.
	100° At surface of contact.	100° After agitation.	100° At surface of contact.	100° At surface of contact.	100° After agitation.	100° At surface of contact.	100° At surface of contact.	100° After agitation.					
Phenol	orange yellow	faint orange yellow	fine violet, below yellow	same, but less decided	helio- trope	yellow brown, turning to black	brown yellow	light brown	nil	faint orange yellow	yellow	nil	
α -Naphthol	brown yellow	yellow	brown yellow	brown yellow, slightly fluores- cent	citron yellow	violet red	dark red, blue above	fugitive, violet	brown yellow, slightly fluor- escent	yellow	orange red	brown yellow	
β -Naphthol	brilliant yellow, fluores- cent	yellow, fluores- cent	fine yellow, fluores- cent	yellow, fluores- cent	yellow, slightly fluores- cent	fine currant red as acid falls, green-brown afterwards	carmine	yellowish	orange yellow, fluorescent	yellow, no fluores- cence	crimson, milky tur- bidity	fine yellow, fluores- cent	
Resorcinol	yellow	yellow	crimson, blue above	faint violet red	rose	greenish brown above	yellow	brown yellow, change- able	salmon rose	yellow	orange yellow	yellow, with green marbling	
Hydroquinone	orange yellow	orange yellow	orange yellow	orange yellow	brown yellow	brown yellow almost nil	almost nil	slightly bistre	orange yellow	orange yellow	orange yellow	orange yellow	
Pyrogallol	orange yellow, rose above	same, but weaker	orange red, violet above	violet	salmon rose	yellow, quickly turning black	yellow, violet above	violet red	faint brown yellow	orange yellow	orange yellow	orange yellow	
Phloroglucinol	yellow	yellow	poppy red	yellow	yellow	orange red, green above	greenish yellow	violet red	brown yellow	orange yellow	orange red	golden yellow	
Guaiacol	faint orange yellow	faint orange yellow	brown yellow, red	faint orange yellow	salmon	brown yellow, black	brown yellow	red brown	currant red, yellow	orange yellow	orange yellow	red yellow	
Thymol	orange yellow	very slightly yellow	brown yellow, blue above	very slightly yellow, blue above	blue	orange yellow, greenish fluo- rescence	orange yellow	violet	yellow, car- mine above	yellow	orange yellow	pale yellow	
Phenyl Salic.	slightly orange yellow	very slightly yellow	brown yellow	fine violet	helio- trope	brown yellow, black	yellow	nil	very slightly coloured, milky turbidity	very slightly yellow	orange yellow	very slightly yellow	
Gallie Acid	nil	nil	slightly yellow	almost nil	slightly yellow	nil	nil	nil	yellow, turning green, fine blue shade above	nil	nil	nil	
Camphor	very faint yellow	very faint yellow	yellow	very faint yellow	old-wine yellow	brown red, blue above	violet red	violet	very faint yellow	very faint yellow	very faint yellow	yellow	

aldehyde under examination, and 1 c.c. of sulphuric acid are employed, and the resulting shades of colour compared with solutions of Bismark brown standardized to the colours given by acetic aldehyde solutions of known strength in response to the same test. In the case of phloroglucin the standard colour solutions are prepared from anilin yellow.

Should the sulphuric acid contain nitrous impurities, accessory reactions will be produced, resorcin, for example, forming a deep blue colour at the surface of contact, surmounted by a fine red tinge, whilst the acid layer turns pale-green.

In applying the test to phenols ordinary phenol may be detected by acrolein (heliotrope). α -Naphthol by furfural violet-red, β -naphthol by benzoic aldehyde, and so on. By employing the aldehydes in a 0.1 per cent. solution the colour reactions are readily differentiated.

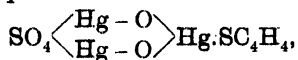
C. S.

A Method for the Separation of Methylamines. M. Delépine. (*Bull. Soc. Chim.*, 1896, 701-704.)—The method depends on the fact that formaldehyde combines with mono- and di-methylamines forming compounds with high boiling-points, but does not combine with tri-methylamine. With mono-methylamine it forms the compound $\text{CH}_2\text{:N.CH}_3$ (B.P. 166°C.), and with di-methylamine yields either $\text{CH}_2\text{:[N(CH}_3)_2]_2}$ or $\text{HO.CH}_2\text{N(CH}_3)_2$ both boiling at $80\text{--}85^\circ \text{C.}$ Since tri-methylamine boils at 9°C. the fractions can be readily separated. To recover the bases from the fractions the condensation products may either be saturated in aqueous solution with picric acid, when, on evaporation, the picrates of mono- or di-methylamine crystallize out, the former melting at 207°C. and the latter at 156°C. Or they may be converted into hydrochlorides by the action of hydrochloric acid and alcohol, and evaporation of the solutions. Mono-methylamine hydrochloride, which crystallizes in plates, melts at $210\text{--}220^\circ \text{C.}$, whilst di-methylamine yields prisms (M.P. 171°C.) which are much more soluble in water or alcohol than the hydrochlorides of the other two bases. Bismuth iodide may also be used as a means of separating the base from the fraction boiling at 166°C. , methylamine forming red hexagonal crystals— $(\text{CH}_3\text{NHI})_3 \cdot 2\text{BiI}_3$ —which, on boiling with potash, yield the base.

Nessler's solution is a test for the purity of tri-methylamine, since with mono-methylamine it gives a yellow insoluble precipitate, while the di- and tri-methylamines yield white precipitates soluble in water.

C. A. M.

Volumetric Estimation of Thiophen in Benzene. G. Denigès. (*Bull. Soc. Chim.*, 1896, 1064, 1065.)—2 c.c. of the benzene are placed in a 60 c.c. flask with 30 c.c. of methyl alcohol free from acetone, and 10 c.c. of mercuric sulphate solution rapidly added. The latter is prepared by mixing red mercuric oxide 50 grammes, sulphuric acid 200 c.c., and water 1,000 c.c. The stoppered flask is left for about 20 minutes, after which the liquid is filtered from the insoluble compound



which is formed under these conditions. 21 c.c. of the filtrate (= 1 c.c. of benzene) are placed in a litre flask with 350 c.c. of water, 15 c.c. of ammonia solution, 10 c.c.

of potassium cyanide (equivalent to N/10 silver nitrate) and 5 or 6 drops of a 20 per cent. solution of potassium iodide, and the whole well shaken. When perfectly clear, slight heat being applied if necessary, decinormal silver nitrate solution is added until there is a permanent turbidity. The amount of thiophen, x , in 1 litre of benzene can then be calculated by the formula $x = (n - 0.3 \text{ c.c.}) \times 2.80$, where n = the number of c.c. of silver nitrate solution (Cf. ANALYST, xx. 188, and this volume, page 303).

C. A. M.

A New Unsaturated Fatty Acid. A. Hebert. (*Bull. Soc. Chim.*, 1896, xv., 941-945.)—This acid, to which the author has given the name of *isanic acid*, is obtained by fractionally precipitating as barium salts the fatty acids obtained from I'Sano seed oil. It crystallizes from ether in foliated crystals, and when purified by recrystallization melts at 41° C. It is easily soluble in strong alcohol, ether, chloroform, benzene, acetone, methyl alcohol and petroleum spirit, and is extremely sensitive to the action of air, rapidly absorbing oxygen and assuming a rose tint. Light also appears to play some part in the change. Its percentage composition is carbon 76.63; hydrogen 9.30; oxygen 14.07—results which correspond to the general formula $C_nH_{2n-8}O_2$; n in this case being equal to 14. The molecular weight as determined by Raoult's method approximated closely to 220, corresponding to the formula $C_{14}H_{20}O_2$. Attempts were made to add hydrogen by heating with hydriodic acid in a sealed tube, but only products of advanced decomposition with traces of a liquid fatty acid were obtained. The acid absorbs two molecules of iodine.

The ammonium salt is soluble in water and can be crystallized from that solvent, the crystals on exposure to air becoming first rose and then blue. The barium salt is a white powder and the lead salt an amorphous yellow powder, which dissolves in ether like the lead salts of the better known unsaturated fatty acids.

C. A. M.

Measurement of Rancidity of Fats other than Butter. A. Scala. (*Staz. Sper. Ag. Ital.*, xxviii., 733.)—Finding that the determination of the free acid in oils and fat was not an absolute measure of rancidity, the author proposes the use of the Reichert-Wollny method for this purpose; Bornemann, Arata, and himself have previously called attention to the invariable presence of volatile acids in rancid fats.

Lard, olive-oil, and stearin were exposed in flat basins, covered by large dishes, to the action of air and sunlight; every 15 days a determination of the total volatile acids by the Reichert-Wollny method, and the free volatile acids by distillation with water (5 grammes fat + 140 c.c. water; 110 c.c. distilled) were determined.

	LARD.		OLIVE-OIL.		STEARIN.	
	Total vol. acids.	Free vol. acids.	Total vol. acids.	Free vol. acids.	Total vol. acids.	Free vol. acids.
Fresh ...	0	0	0	0	0	0
15 days ...	0.6	0	0.2	0	0.8	0
30	0.8	0.2	1.0	0	0.9	0
45	2.5	1.5	1.5	0	1.8	0.4
60	4.0	1.6	2.8	0.4	2.0	0.6
75	5.2	2.3	3.2	0.8	2.3	0.8
90	7.8	4.6	3.3	1.1	2.5	1.8

H. D. R.

With the oil from linseed-cake, however, the results were considerably lower, only one sample giving an iodine number of 184, whilst the others were 166, 167, 168, etc. None of the oil extracted from the linseed-cake was rancid or soluble to any extent in acetic acid, so that alteration of the oil was not the cause of the low figures. In order to decide whether the presence of foreign seeds lowered the iodine number, the oil was extracted from mixtures of pure linseed with the foreign seeds usually met with in linseed, and the iodine number determined.

	Iodine No
Linseed, 100 per cent.	187
Rape seed (<i>Brassica campestris</i>) 100 per cent.	101
Cameline seed (<i>Camellina sativa</i>) 100 per cent.	146
90 per cent. linseed and 10 per cent. cameline seed	179
85 " " 15 " " " " " " " "	172
80 " " 20 " " " " " " "	170
90 " " 10 " rape seed " " " " "	173
85 " " 11 " " and 4 per cent. cameline seed	174
90 " " 7 " " " 3 " " "	176

Finally they tested the oil from the cakes with alcoholic silver nitrate solution (Brullé's test), and with several obtained an unmistakable reaction which they ascribed to the presence of cotton-seed oil. With the oil from one of the cakes the reagent produced a peculiar fatty mass unlike anything obtained with pure linseed-oil or with oil from the above-described mixtures. The "oil" from another cake was stiff at the ordinary temperature and had a white, horn-like appearance. The conclusion arrived at was that, in addition to the microscopical examination of linseed-cake the iodine number of the extracted fat should always be determined, and where this is low, and at the same time the amount of foreign seeds small, special search should be made for cotton-seed oil and other oils.

C. A. M.

The Composition of "Driers." M. Weger. (*Zeit. angew. Chem.*, 1896, 531-536).—The author considers that Amsel has based too much on incomplete theory in his recent communication on this subject (*ANALYST*, xxi., 261). The formulæ for the colophony acids are not yet settled, and the adoption of one or the other makes a difference of 2 per cent. in the amount of lead required by theory for the lead salt. Although Maly once found a stated percentage of abietic acid, etc., in a sample of colophony, a hundred samples might be examined without finding one of a precisely similar composition. Even in one and the same specimen the author has found considerable difference between the upper and lower portions in the proportion of the different acids and the degree of anhydride formation, as well as in the amount of water and ethereal oil. Moreover, variations in the amount of metal may be produced during the manufacture, and in the case of melted manganese resinate it is doubtful whether the metal is present as manganous or manganic oxide, or as a mixture of both. In precipitated "driers" the possibility of the formation of basic salts is to be taken into account.

The requirements for a soluble "drier" in practice are that it must be completely soluble, and that the metal must be in combination and not partially suspended as oxide. The estimation of the total amount of metal is therefore no criterion of the value of a product, since the suspended matters in many commercial siccatives (PbO , Mn_2O_3 , CaO , etc.) are not only valueless, but are distinctly harmful to the varnish.

The best solvents for testing a preparation are ether and (in the case of lead resinate) chloroform, the solubility of a siccative in these corresponding with its solubility in linseed-oil, with the difference that no heat is required. Therefore when a product is insoluble in cold ether or chloroform, it is also insoluble in moderately hot linseed-oil, and is worthless. Only lead and manganese are used for "driers," although zinc, calcium and barium resinates are mixed with copal in commerce, but if no lead or manganese be present the preparations are of no use in varnish manufacture. The higher the amount of soluble lead or manganese, in the absence of insoluble matter, in a siccative, the less is required to be added to a linseed-oil.

Lead siccatives are hardly used, the ordinary preparations being manganese resinate, lead and manganese resinate, manganese linoleate* and lead and manganese linoleate. As regards their composition, the author, from the examination of hundreds of samples, finds that the soluble manganese in fused resinates seldom exceeds 3.2 per cent. (manganic oxide), whilst in precipitated resinates it is seldom over 6, or in exceptional cases 7, per cent. (manganous oxide). Good preparations of fused manganese linoleate have from 9 to 9.5 per cent. of soluble manganese, though the author has found as much as 11 per cent., an amount which points either to a deep-seated change during the heating or to the formation of basic salts. Precipitated manganese linoleate does not occur in commerce. The preparations most used are the fused lead-manganese resinates, and in these the most suitable relation of lead to manganese appears to be as 5 : 1. The products of one of the best-known firms are fairly concordant in this respect, having 8 to 9 per cent. of soluble lead, with 1.5 to 2 per cent. of soluble manganese. Lead-manganese linoleate, which is not much used, comes into the market with very varying composition. Higher percentages of soluble

* *Linoleate* here means the metallic compound of the mixed linseed-oil acids.

metals than the above-mentioned are invariably accompanied by insoluble metallic compounds, which are, in fact, only completely absent in very few preparations. The fact that many samples contain a considerable quantity of insoluble matter (Mn_2O_3 , MnCO_3 , etc.) does not prevent free resin and linseed-oil being present, and most of the fused preparations in the market contain both. Falsification of resinsates with resin is practically non-existent, notwithstanding Amsel's supposition, nor could much profit be derived from the practice; but the case is otherwise with linseed-oil siccatives, where the addition of resin would pay, although the author has not yet met with it.

With regard to the use of soluble siccatives, the quantities required for the preparation of a good varnish are: melted manganese resinate 2 to 3 per cent.; melted lead-manganese resinate 2 to 3 per cent.; melted manganese linoleate 1 per cent.; precipitated manganese resinate 1 to $1\frac{1}{2}$ per cent. The siccative is either put directly into the linseed-oil heated to about 120°C . (not higher than 150°), or preferably 1 part of the siccative is dissolved in 2 parts of linseed-oil at 120° , and the mixture stirred into the main bulk. The oil can be oxidized either before or after the addition of the "drier." For clear varnish lead-manganese resinate is used, and where all separation is to be avoided, melted manganese resinate. For the preparation of turpentine-oil siccatives melted manganese resinate, manganese linoleate, and lead-manganese linoleate are employed dissolved in the turpentine-oil in the proportion of 1 : 2 or 2 : 3.

In the analytical examination of soluble siccatives the organic matter is first burnt off and the lead and manganese in the ash determined. It is useless to weigh the total ash, since resinsates often contain sand. If, after the removal of lead, calcium be present to any extent, the manganese and calcium are determined together in neutral solution as carbonates, the manganese titrated and the calcium found by difference. The insoluble lead and manganese are then determined by dissolving a fresh portion of the siccative in ether or chloroform, filtering, washing, igniting, etc. The soluble manganese is determined by the difference between this result and that of the total manganese, and the result may be controlled by determining the soluble manganese in an aliquot part of the filtrate. The soluble lead must be determined by difference, since the chloroform cannot be completely evaporated from the resinate solution, traces remaining except at red heat, when most of the lead volatilizes with it as lead chloride.

C. A. M.

The Estimation of Phenol in Soaps and Disinfectants. H. Fresenius and C. J. S. Makin. (*Zeit. anal. Chem.*, 1896, xxxv., 325-334.)—The estimation of phenol in aqueous solutions, or in crude carbolic acid, presents no special difficulty, but the case is often otherwise when the phenol is mixed with soap. Of the methods hitherto employed, the authors prefer that of Low, in which the fatty acids are liberated and the phenol determined in the filtrate either gravimetrically, as tri-bromo-phenol, or volumetrically by Koppeschaar's process (*Zeit. anal. Chem.*, xv., 233). The method now described is based on the fact that phenol is volatile with steam, and can be determined in the distillate, preferably by Tóth's modification of Koppeschaar's process (*Zeit. anal. Chem.*, xxv., 160).

The solutions required are : (1) Sodium thiosulphate containing 9.763 grammes per litre. This is standardized on iodine, and from the result the equivalent of bromine calculated. (2) A bromine solution containing 2.040 grammes of sodium bromate and 6.959 grammes of sodium bromide per litre. (3) Clear starch solution. (4) A solution of potassium iodide containing 1.25 grammes, which is freshly prepared for each determination.

The bromine solution is standardized on the thiosulphate solution by shaking 25 c.c. of the former with 5 c.c. of hydrochloric acid for a few minutes in a stoppered flask, adding 1.25 grammes of potassium iodide, and titrating with the thiosulphate. The amount of bromine contained in 1 c.c. is thus determined.

In determining the quantity of phenol in a solution an excess of the bromine solution is added with 5 c.c. of hydrochloric acid, and the stoppered flask shaken for 30 minutes. A freshly-prepared solution of potassium iodide (1.25 grammes in 30 c.c.) is then added, and after standing for 12 hours the liberated iodine titrated with standard thiosulphate. The amount of bromine used is thus obtained, and from that the equivalent quantity of phenol by the proportion $6\text{Br} : \text{C}_6\text{H}_5\text{OH}$ (479.70 : 94).

A solution of phenol of known strength was titrated directly by this method, and after distillation either over a Bunsen flame or by means of a rapid current of steam; concordant results were obtained in each case. Next, mixtures of the phenol solution and soap were prepared, the fatty acid separated from the latter by means of dilute sulphuric acid and the phenol distilled in a current of steam. In this case the results were about 1 per cent. too high, which was attributed to the action of the bromine on traces of fatty acids in the distillate. Blank experiments were therefore made with soap containing no phenol under exactly similar conditions, and the results being deducted from the previous figures, gave the amount of phenol required by theory. Several carbolie soaps were tested in this way, and in one there was found (a) 1.503, (b) 1.665 per cent.; and in another (a) 3.38, (b) 3.51 per cent. of phenol. In a sample of phenol soap which, according to the preparation formula, contained 1.738 per cent. of phenol, the analysis gave 1.643 per cent.

In examining disinfecting powders, about 0.5 gramme is taken, water added, with about 50 c.c. of concentrated hydrochloric acid, and the phenol distilled over by means of a rapid current of steam, and titrated in the distillate as described above.

C. A. M.

INORGANIC ANALYSIS.

The Determination of Sulphuric Acid, or of Barium. J. Edmunds. (*Chem. News*, lxxiv., 187.)—The author proposes to estimate sulphuric acid by precipitation with an excess of barium nitrate, the residual barium being precipitated by excess of neutral potassium chromate, and the residual chromate by silver nitrate in excess, the mixture being well shaken after each addition. It is then filtered, and an aliquot part of the filtrate titrated for its residual silver nitrate, potassium chromate being used as indicator. The total of the residual silver nitrate, deducted from the silver nitrate originally employed, gives the equivalent of the sulphuric acid.

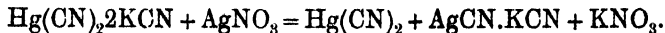
Each precipitate is thrown down in the presence of an ample excess of precipitant, and the minute quantity of silver chromate which would remain in solution in a filtrate charged with certain salts is compensated for by using the potassium chromate indicator in the titration of the residual silver.

Haloid radicles and other precipitants of silver are best estimated by a preliminary titration and allowed for in the final titration. A preliminary volume of the solution is shaken with $\frac{N}{10}$ silver nitrate in excess, the precipitate filtered off, and the residual silver titrated by means of thiocyanate with ferric sulphate.

In determining barium the solution is shaken with an excess of $\frac{N}{10}$ neutral sulphate, the amount of the excess being afterwards ascertained by the process above described. Freedom from other substances, such as lead or strontium, which precipitate sulphuric acid, is presupposed.

C. A. M.

General Method for the Estimation of Mercury. G. Denigès. (*Bull. Soc. Chim.*, 1896, 862-871.)—The method is based on the facts that when an excess of potassium cyanide is added to the solution of a mercuric salt, the compound $\text{Hg}(\text{CN})_2\text{KCN}$ is formed, and that on adding ammonia and running in silver nitrate solution, this double salt is decomposed :



A solution of potassium iodide is used as indicator, and the completion of the reaction is shown by a permanent opalescence due to silver iodide.

The solutions required are decinormal silver nitrate, an equivalent solution of potassium cyanide, and a 20 per cent. solution of potassium iodide. In making an estimation, 10 c.c. of ammonium hydrate are added to the solution of the mercuric salt, then an excess of potassium cyanide solution, and after the addition of a few drops of potassium iodide, the silver nitrate run in until the opalescence remains permanent. The difference between the number of c.c. of potassium cyanide taken, and the number of c.c. of silver nitrate used, gives the amount a of decinormal silver nitrate corresponding to the mercury in the solution.

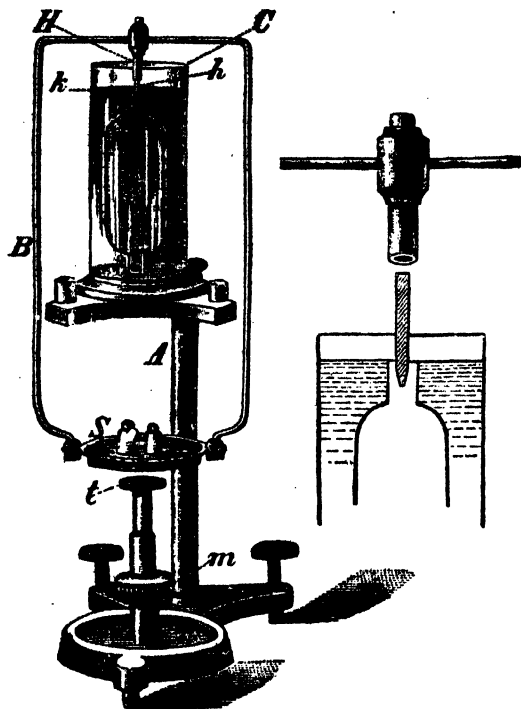
In practice the reaction is never completed according to the equation, a small quantity of the double cyanide remaining undecomposed, and for very accurate work a correction must be made. This may be calculated by the following rules : (1) When a is between 0 and 5.5, the true value, x , $= a \times 0.96$. (2) When a lies between 5.5 and 9.5, $x = (a \times 1.04) - 0.45$. The amount of mercury is obtained by multiplying x by 0.020 grammes.

It is stated that the estimation may be made in the presence of strong mineral acids, that alkalis do not affect it, and that it is capable of very general application.

C. A. M.

APPARATUS.

An Improved Hydrometer. T. Lohnstein. (*Chem. Zeit.*, 1896, xx., 559).—This instrument is essentially a form of the old Fahrenheit hydrometer, so modified as to obviate the disturbing influence of capillarity. The principle is as follows: If a glass tube, loaded in the usual manner so as to float vertically, is cut off at the



level of the liquid in which it is immersed at right angles to its major axis, the effect of some considerable addition to its weight is simply to alter the meniscus from concave to convex without causing the instrument to sink. If the instrument does not float quite vertically, the true position will be when the concavity of the meniscus on the one side is equalled by the convexity on the other, while the two points midway are absolutely at the level of the liquid.

The general form of the apparatus is shown in the figure. G is the hydrometer bulb, of such a size as to displace 50 c.c. of water at 15° C. h is a glass rod ground and cemented into the aperture of G, while at the other end it slips loosely into the tube H of the brass scale pan B-S. t and m are parts of an ordinary releasing gear to prevent overloading. The machine is adjusted so that

with the pan in position and empty G assumes its "Archimedean position" (as shown in the enlarged plan) in a liquid having the specific gravity 0.7 at 15° C. Seventeen brass weights are provided corresponding to increases of density (over 0.7) of 0.5, 0.3, 0.2, 0.1, 0.05, . . . 0.0001, so that by adding together the values of all the weights required to make G float normally, the specific gravity of the liquid under examination is given directly.

When only small variations of density are expected, the apparatus may be simplified by removing the stirrup-pan and substituting the flat table of Fahrenheit; or the vessel may be constructed in the form of an inverted cone, loaded at the apex to give it a still greater range, with a table on the top for small weights, and two or three riders equal to 0.3, 0.6, and 0.9 respectively.

The author claims to get figures exact to the fifth place of decimals if proper care as to temperature, etc., be taken.

F. H. L.

LEGAL.

IMPORTANT DECISION WITH REFERENCE TO MILK CERTIFICATES.
QUEEN'S BENCH DIVISION.

(Before MR. JUSTICE GRANTHAM and MR. JUSTICE KENNEDY.)

BRIDGE v. HOWARD.

(Reprinted from the "Times" of October 29, 1896.)

This was a special case stated by two justices for Middlesex in regard to a milk adulteration case, and raised a point as to the sufficiency of the analyst's certificate.

Mr. EARLE appeared on behalf of the appellant, the Public Analyst, and said the case arose in consequence of the decision of the Divisional Court in *Fortune v. Hanson* (1896, 1 Q.B., 203), when a certificate was held bad which stated only that the sample "contained the percentage of foreign ingredients as under 5 per cent. of added water." The certificate in this case was in the following words:

"I, the undersigned, Public Analyst for the county of Middlesex, do hereby certify that I received on March 26, 1896, a sample of new milk (E.M. 159) for analysis (which then weighed 6 oz.), and have analysed the same, and declare the result of my analysis to be as follows: I am of opinion that it contains the parts as under—milk, 94 per cent.; added water, 6 per cent. This opinion is based on the fact that the sample contained 7.97 per cent. solids-not-fat, whereas genuine milk contains not less than 8.5 per cent. solids-not-fat. The sample had undergone no change which would interfere with the analysis."

At the hearing of an information on April 24 against the seller of the sample of milk, this certificate was tendered by the appellant, but it was objected, on behalf of the respondent, that it was inadmissible on the ground that it did not state specifically, as the result of the analysis, the parts contained in the sample analysed. The hearing was then adjourned, and on the rehearing the appellant contended that the certificate was valid and sufficient, and also tendered the oral evidence of the Public Analyst, who was present, as to the constituent parts of the sample, and as to the inutility of stating specifically the amount of the water and other constituent parts of the milk, having regard to the usual and scientific method of analysis, and as to the proper manner of analysing milk. The respondent objected, on the ground that proceedings for the recovery of penalties can only be maintained when a certificate in compliance with the statute has been given, and that the analyst could not by oral evidence supplement his certificate. The justices, being of opinion that the certificate was insufficient, as not setting out specific quantities of all the constituent parts of the sample, and also that the proceedings could not be maintained in the absence of a certificate setting out the said quantities specifically, declined to hear the oral evidence, and dismissed the information.

Mr. EARLE contended that the certificate was sufficient, as it gave the analyst's reasons for saying that a certain proportion of water had been added. The justices had the materials afforded them to enable them to say whether they adopted the conclusion arrived at by the analyst, and in this respect there was a difference from the certificate in *Fortune v. Hanson*. It was proved that there was a certain deficiency of solids in the sample. Therefore a certain quantity of foreign ingredient must have been added; and inasmuch as the sample had undergone no change, the foreign ingredient must be water. The analyst had made an analysis on a different principle to that contemplated in the case referred to, and he considered that it would serve no useful purpose to state the total amount of water in the milk, both natural and added. On the second point—namely, the refusal of the justices to hear evidence—he admitted that if the certificate was not in accordance with the Act they were justified in refusing evidence. The point was, therefore, the same as to that part of the case as in the other.

The COURT upheld the validity of the analysis (certificate).

Mr. JUSTICE GRANTHAM said the justices were wrong in rejecting the certificate. The analyst here had done enough in setting out the amount of solid substance which he found in the sample, and basing his decision thereon. It was hopeless to go into the question of how much water there was in the milk altogether. In some cases rich milk which had been adulterated with added water contained less water than perfectly pure milk of a poor class. The analyst had stated the principle on which he came to his conclusion. It was simply a rule of three sum, and, in fact, he had rather understated matters when he gave 6 per cent. as the amount of added water.

Mr. JUSTICE KENNEDY, having been one of the Judges in *Fortune v. Hanson*, desired to add that he was still entirely of opinion that a similar certificate to the one in that case would be bad. It merely contained a statement of opinion that a certain percentage of water had been added, and it did not give the ground for that opinion so as to enable the justices or the party accused to test the accuracy of it. He thought it fairer that the proportionate parts should be set out, and as water was an ingredient in all milk, more was required than a statement that there was water added. Here the analyst not only stated that 6 per cent. of water had been added, but he gave scientific reasons for that statement. That made the difference between the certificate in this case and in the other. He concurred in thinking that this certificate was sufficient. (For report of the case of *Fortune v. Hanson* see the ANALYST, this volume, p. 53.)

REVIEWS.

ALIMENTOS Y BEBIDAS, INVESTIGACION DE SUS ALTERACIONES Y FALSIFICACIONES, por el Dr. CÉSAR CHICOTE.

THIS is a goodly octavo of 734 pages, from the pen of the principal of the municipal laboratory at San Sebastian, and probably forms a good outline of what was known in Spain at the end of 1893 on the subject of the adulteration of food and drink. It is modelled very much on the French style, and ignores all direct reference to the German, English and American literature on adulteration. Indeed, in the three appended pages of bibliography only one English work, Sutton's "Manual of Volumetric Analysis," is directly quoted, and this in the French translation. French, Portuguese and Italian works are freely quoted. Subject to these reservations, the information is copious, and, as might be expected, wines are treated of in an elaborate manner. The illustrations—mostly French—are excellent, and we congratulate our Spanish confrère on his creditable production. T. S.

THE BOOK OF THE DAIRY, TRANSLATED FROM THE GERMAN OF PROFESSOR FLEISCHMANN. BY C. M. ATKMAN AND R. P. WRIGHT. (London, 1896: Blackie and Son.) xxiv+344. Price 10s. 6d.

"The Book of the Dairy" differs from most works on milk, in that it partakes more of the nature of a monograph than of a compilation; though the author has not hesitated to make judicious use of the researches of others, the groundwork of the book is solid fact, the result of his laborious investigations. To say that the author is Fleischmann is very nearly a sufficient critique; yet the translation has faults.

Of what practical use is it to the British dairy farmer to know the profit yielded by different treatments of milk in pfennige per kilo? Why should dairy utensils in

use in Germany only be mentioned in the text, while the illustrations refer to those of British make, which are otherwise ignored? Why are centigrade degrees, kilos, and litres used in a book designed for men who think and work in Fahrenheit degrees, pounds, and gallons? Had the translators but seen their way to Anglicize to a greater extent, the book would have been really useful, instead of merely instructive.

The chemical portion of the book is clearly and concisely written, and on the whole very exact, but is distinctly behind the times; this is perhaps due to the delay of translation, the work appearing simultaneously with that of a second and enlarged German edition.

The analytical part is somewhat weak; for example the Adams' method is given second place to the process of extraction of fat with sand; the essential point of this beautiful method is missed, when it is directed to place as much as 8 to 10 grammes of milk on a coil. The method for total solids, too, is distinctly inferior to that used in England.

It is greatly to be regretted that analyses have been "cooked" to make them add up to 100.00; for instance, on page 269, an analysis of "curd-whey" duly adds up to this figure, and includes 4.87 per cent. milk-sugar; in the original analysis, which added up to 99.506, the milk-sugar was returned as 4.377 per cent.

It is surely time that the analyses of condensed milk on pp. 284, 285, which date from 1868-1878, were replaced by figures showing the composition of the modern product. The same applies to Gerber's analyses of commercial milk-sugar (made in 1878), which show only 92 and 86 per cent. of milk-sugar; to-day 99.7 per cent. is a more usual figure. One of Dr. Gerber's analyses is remarkable for adding up to 99.99 per cent.

Among errors which are misleading, the apparent contradiction between the statement (page 16) that the specific gravity of perfectly fresh milk is *higher* than that of milk after standing, and that on page 68 that it is *lower*, and the statement that the lacto-butyrometer cannot be used for milk containing *more* than 1.339 per cent. fat (instead of *less*), may be mentioned; also the use of the terms "absorptive capacity for heat," "latent heat" (page 13), and "specific heat" (page 117), as identical in meaning.

We find in the directions that after filtering "in the presence of a weak diluted atmosphere" a glass tube is to be "stretched and bent on a holder downwards" (page 85), and the confusion between of the terms "cheese-milk" (Käse milch) and "whey" (molken) (page 269) does not reflect credit on the translators.

There is a quite unintelligible passage on page 28, referring to the amount of phosphoric acid derived from the casein, where the translators do not seem to have grasped the author's meaning.

The bulk of the information is very correct; figures that are usually misstated, e.g., the density of solid milk-sugar 1.545 (page 95), are here correctly given.

Despite the fact that much of the information is out of date, and much essentially Teutonic, this work can be highly recommended to public analysts and consulting chemists as affording an insight into dairy practice.

H. D. R.

A SYSTEMATIC HANDBOOK OF VOLUMETRIC ANALYSIS. By FRANCIS SUTTON, F.I.C., F.C.S. Seventh edition, enlarged and improved. London, J. and A. Churchill.

All analysts will welcome the appearance of the new edition of Mr. Sutton's well-known work, which brings the subject with which his name has been so long associated well up to date.

Among the sections which are either entirely new, or have been altered and added to, are those on the Calibration of Instruments, the Kjeldahl Process, Boric Acid, Hydrofluoric Acid and Fluorides, Cyanogen and Cyanides, Phosphoric Acid, Sugar, Tannin, Zinc, Urine, etc.

In his preface the author states that, as respects the volumetric method as applied to any organic substances, and the action of modern indicators in such work, nothing has been attempted, mainly because the subject comes specially within the scope of Allen's "Commercial Organic Analysis." Mr. Sutton states that he has availed himself in some instances "of the excellent abstracts of original papers now being published in THE ANALYST, which reflect great credit on the present management in this department."

In the case of a work which has been so long known and highly appreciated as that under review, and which has reached its seventh edition, there is little room for criticism. The arrangement of the subject-matter is familiar to every chemist, and at worst any particular article wanted can be found by the aid of the index. Throughout, one is struck with the author's remarkable powers of assimilation, processes of all kinds being described in their proper place, and in Mr. Sutton's well-known terse and lucid style. The article on Cyanides contains the most recent processes for examining the cyanide liquors of gold works.

Amongst collaborators, the author makes mention of, and expresses his thanks to, Mr. W. B. Giles for an original article on the Estimation of Hydrofluoric Acid, and for the benefit of his long practical experience in the examination of sulphur compounds and phosphoric acid; to Mr. J. W. Westmoreland for great services rendered in the articles on Copper, Iron, and Manganese; and to Dr. James Edmunds for suggestions on Urinary Analysis which the author believes to be of considerable practical value.

The nomenclature adopted is mainly the same as in previous editions. The section on Water Analysis has been added to in various respects, and is now one of the most complete treatises on the subject in the language; while in the chapter on the Volumetric Analysis of Gases the author has placed on record a number of recent improvements.

The whole work now covers 487 closely-printed pages, and is a monument of carefully-collated information. "Sutton" has long been one of the most constant companions of analytical chemists of every class, and the new edition fully maintains the reputation of the author for excellence of work in every respect.

A. H. A.

THE ANALYST.

DECEMBER, 1896.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday, November 4, in the Chemical Society's Rooms, Burlington House, the President (Dr. Stevenson) occupying the chair. The minutes of the previous meeting were read and confirmed.

The following gentlemen were elected as members of the Society: Frederick James Montague Page, B.Sc. (Lond.), Assoc. R.S.M., F.I.C., Lecturer on Chemistry at London Hospital, and Charles George Matthews, F.I.C., Analyst and Consulting Chemist, 58, Charing Cross, London.

Mr. David J. Morgan, of Swansea, was proposed for election as member; and Mr. Percy S. Marshall (assistant to Mr. Jarmain), Mr. Percy T. Adams (assistant to Dr. Adams), and Mr. Louis C. Deverell (assistant to Mr. Kitto), were proposed for election as associates.

The following papers were read: "Note on Ginger," by Thomas P. Blunt; "The Determination of Stearic Acid in Fats," by Otto Hehner and C. A. Mitchell, B.A.; "Further Note on Lead in Canadian Cheese," by F. Wallis Stoddart.

NOTE ON GINGER.

By THOMAS P. BLUNT, M.A., F.I.C.

(Read at the Meeting, November 4, 1896.)

THE following account of a case tried at Newport, Salop, last September, may be of interest to the members of this Society.

I condemned a sample of powdered ginger as adulterated with not less than 25 per cent. of the exhausted root, the data upon which I relied being the following:

Total ash	2.74 per cent.
Soluble	1.24 "
Cold water extract	6.2 "

The wholesale house from which the sample had been bought by the retailer undertook full responsibility for it, and provided the defence with a solicitor.

An expert was brought forward, who said that he had examined the portion of the sample left with the vendor, and "found it to be genuine ginger of high quality, and absolutely free from spent ginger. Every particle appeared to be genuine. If there had been any spent ginger, he should have detected it by the changed shape of the starch granules." (I am quoting from the report of a local paper, for the

substantial accuracy of which I can vouch.) "He subjected it to a chemical test, and found it contained 6 per cent. of resin and 1.25 of essential oil, which were perfectly normal quantities. . . . The microscope was the truest test, the other"—soluble ash and cold water extract—"was only inference. He agreed with Mr. Blunt's figures to some extent, but his conclusions were wrong."

This gentleman said that his experience in the analysis of ginger extended to some hundreds of cases a year, and as there is much in his evidence which is entirely at variance with the statements of other analysts who have made a special study of ginger, I bring the subject forward to-night in the hope of eliciting a free expression of opinion, and generally of throwing light upon it.

As regards any change in the starch granules by exhaustion, I produce the accompanying photo-micrographs, kindly taken for me by a friend, representing :

1. Genuine Jamaica ginger of high quality.

2. The same ginger after it has undergone a very thorough process of exhaustion, in the ordinary course of preparation of the stronger tincture of the British Pharmacopœia, the process being supplemented by expulsion of the spirit with distilled water; the whole operation occupied rather more than five days, and left the marc without taste or smell.

- 3 and 4. Mixtures of the two samples as above, in the proportion of one of genuine ginger to one of exhausted (50 per cent. genuine). I think it would puzzle anyone present to pick out the granules "of changed shape" from the mixture, or, indeed, to recognise any change of shape at all in the starch granules of the exhausted sample.

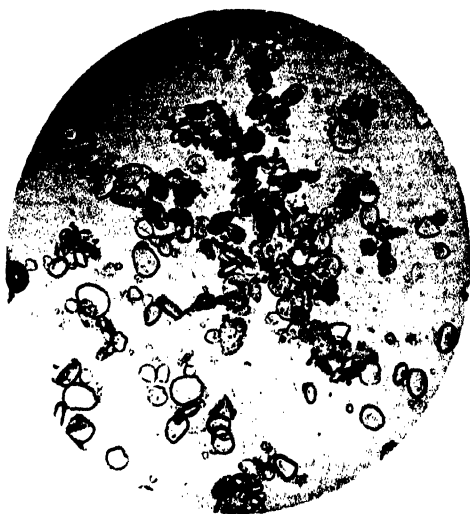
In conclusion, I may say that the case was dismissed by the magistrates on the ground that they were not satisfied that the ginger was adulterated. They refused, however, to allow costs.

DISCUSSION.

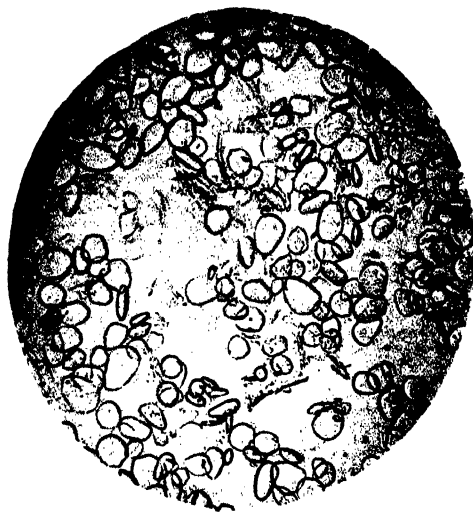
Dr. DYER said that, as it was pretty well known, from the reports which had appeared in the newspapers, that the gentleman who appeared for the defence in this case was Mr. Collingwood Williams, the colleague of Dr. Campbell Brown, joint Public Analyst for Lancashire, he (Dr. Dyer) had considered it courteous to send Dr. Campbell Brown a copy of Mr. Blunt's note, and had suggested that, if he or Mr. Williams could not come to the meeting, they might like to send some contribution to any discussion that might arise. He had received the following communication from Dr. Campbell Brown :

"Dr. Bernard Dyer has asked me to send to the Society of Public Analysts some statement of my views on the analysis of ginger and of spent ginger, and to discuss a paper which is to be read, he tells me, at the next meeting of the Society, and has very kindly taken the trouble to send me a copy of that paper.

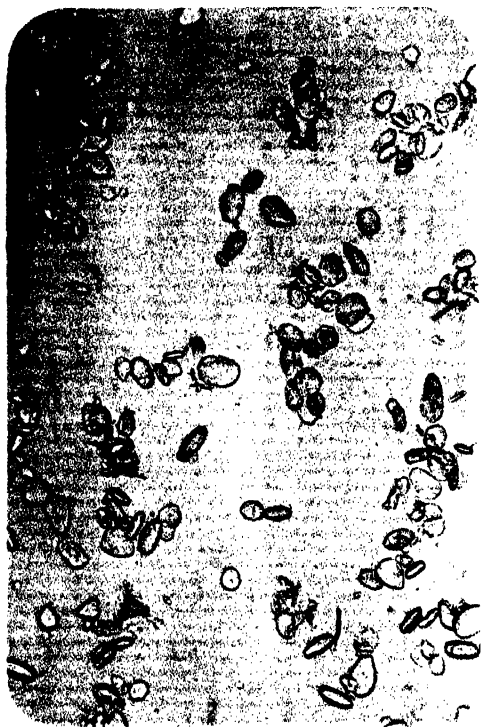
"I am very willing to give briefly the view to which I have been led by twenty-five years' experience, but it would not be proper to discuss or re-try any case in the absence of the persons who are by implication accused of fraud. There are other objections to discussing the paper. The first is the general objection just mentioned.



1.



2.



3.



4.

Secondly, it is always a mistake to discuss newspaper reports; for although reporters do wonderfully well, nevertheless they cater for the amusement of the public, and unless every reporter had a special scientific and expert training, which is absurd, he could not write a condensed report which would be reasonably available for scientific purposes.

"No chemist would maintain that the microscope is the most important or truest test as applied to ginger, but it would be correct to say that the microscopic examination, when it is available, as it often is, has the advantage of affording direct evidence, as distinguished from the circumstantial evidence afforded by chemical analysis.

"Thirdly, the experiments adduced in the paper and the photo-micrographs accompanying it merely prove what no one would dream of denying, that a chemist can by means of alcohol extract ginger without affecting the microscopic appearance of the starch granules. This manifest truth does not affect the question in any way.

"An analysis which would give reliable information regarding spent ginger should include estimation of total ash, ash soluble in water; sometimes a further examination of the ash, alcoholic extract (with such an examination as satisfies the analyst that it is bona-fide ginger extract that he has got), and essential oil (here, again, the analyst should be satisfied that it is the essential oil of ginger); and lastly, taste, smell, and appearance to the naked eye and under the microscope.

"An experienced man will sometimes be able to tell what kind of ginger he is dealing with, and, if so, that will give him important assistance.

"When spent ginger has been extracted by rectified spirit, the amount of alcoholic extract and essential oil will alike be reduced.

"Sometimes this variety of spent ginger is detected by the microscope—sometimes it may not be so; but it is not detectable by the analyses so frequently made, including only total ash, soluble ash, and aqueous extract. When spent ginger has been prepared by means of water or very dilute spirit, both the soluble ash and the alcoholic extract and the essential oil will be reduced, and invariably in commercial spent ginger of this kind a greater or less change in the microscopic appearance has been produced. If, therefore, the microscope fails to show any visible change, it is necessary to consider very carefully the figures for alcoholic extract and essential oil.

"The amount of cold water extract is of very little value as evidence, and, although there is no harm in determining it, analysts should be careful to avoid being misled by these figures; the amount varies very much in different kinds of genuine ginger. The reduction of soluble ash and of total ash, when nothing else is low, may indicate a high class of ginger, and not a fraudulent sample.

"To sum up, the chemical evidence is the most important and truest, but only if the analyst's mind is impressed by the important ingredients rather than by the unimportant ingredients.

"The true place of the microscope is to furnish frequently very valuable confirmation of either positive or negative evidence."

Dr. DYER also read the following communication from Mr. W. F. Keating Stock:

"I am unable to attend the meeting on Wednesday next, but, in view of

what I trust may prove to be a conclusive discussion of the spent-ginger question, I venture to put my experience into your hands in a condensed form, to deal with as you may think proper.

"In the first place, I may mention that I have had a large number of commercial samples of ground ginger through my hands, including, I think, all the different brands on the market, and that, officially, I have been concerned in fourteen cases of adulterated ground ginger where the proceedings were based upon my own certificates, and where in every case the adulteration was proved to the satisfaction of the justices. In all these cases I have relied upon :

"First, the general appearance and physical condition of the sample ; second, the relation of total ash to soluble ash ; third, the relation of potash (determined as chloroplatinate) to the soluble ash ; and fourth, the relation of sulphuric anhydride and calcium oxide to the soluble ash.

"Out of ninety-four samples of genuine commercial ground ginger recently analysed by me, I have found the soluble ash vary between 1.70 per cent. and 3.60 per cent., and the potash between 0.80 per cent. and 1.80 per cent. The lowest soluble ash and potash occurred together, and were found in a very finely bolted Jamaica ginger commanding a high price, whilst the highest soluble ash and potash were found together in a common red ginger.

"In samples containing exhausted ginger I have found the soluble ash varying from 0.20 per cent. to 1.60 per cent., and the potash from 0.02 to 0.80 per cent. The latter figure overlaps that found in high-class Jamaica, but the condition and general characters of the adulterated sample spoke for themselves.

"A question arose in my mind in connection with a recent case of alleged addition of spent ginger, as to whether the high grading of Cochin ginger could lead to such a diminution of soluble ash and potash in the fine product as would cause the latter to approximate to 'spent' in these figures. In order to test this, I roughly ground a large sample of known genuine Cochin ginger-root, and then bolted it carefully through brass wire bolting cloth of 5,000 holes to the square inch (a size in common use in the trade). In this way I got from 100 grammes 45.72 grammes of rough fibrous, and 54.28 grammes of fine. These portions were analysed along with the original sample, and the following results were obtained :

			Rough Fibrous.	Fine.	Whole Sample.
Total ash	4.61	3.46	3.99
Soluble ash	2.30	2.04	2.16
Insoluble ash	2.31	1.42	1.83
Potash	1.03	.96	.99

"These figures, I submit, show conclusively that, whilst the insoluble ash (as might have been expected) mounts up in the coarse portion, the soluble ash and potash are affected but slightly by the process of grading, and thus dispose of the specious argument that 'fine' and 'high quality' gingers approximate in composition to 'spent.'

"I must conclude with a word as to the microscopic characters of spent ginger. After examining some hundreds of samples, I have no hesitation in saying that the microscope utterly fails to distinguish between the starch of 'spent' ginger and that

found in the outer layers of 'washed' and 'bleached' root. And I have within the last fortnight found altered starch granules in samples of whole ginger which I know to be beyond suspicion."

The PRESIDENT (Dr. Stevenson) observed that Mr. Blunt's communication was one of great interest, and the letters from Dr. Campbell Brown and Mr. Stock were equally important. He could not himself pretend to have any special knowledge of the subject, but he was able to say that microscopical examination had never afforded him any conclusive proof as to whether a ginger was genuine or not. As he understood, it was common to submit dirty ginger to a process of washing, the ginger being sent out to be washed, and returned for grinding. It was supposed to possess a higher commercial value after having been washed. Presumptively the washings were used for trade purposes (he believed they were used in making fermented ginger-beer); and the question of course arose whether this process—which he would not say was one intentionally fraudulent, as it was supposed to improve the quality of the ginger, though, at the same time, it did undoubtedly remove some of the soluble constituents—was a legitimate process.

Mr. ALFRED H. ALLEN said that the newspaper reports of this case stated very distinctly that Mr. Collingwood Williams, representing himself and his colleague, Dr. Campbell Brown, gave evidence that they were satisfied, on the basis of the microscopical examination, that there was no particle of exhausted ginger in the sample condemned by Mr. Blunt; and that the defendants' counsel so understood was clear from the fact that, in his opening speech for the defence, he said that the wholesale dealers were always willing to have their samples tested, provided that the microscope test was alone taken as the basis of the examination. He added that those he represented had no confidence in, and placed no reliance upon, any other test than the microscope. This was the statement which was understood by the newspaper reporters to have been made by Mr. Williams in evidence; and it appeared, from correspondence which he (Mr. Allen) had had with Mr. Blunt, that that gentleman had understood Mr. Williams to say the same thing. He (Mr. Allen) had naturally been interested to learn what it was that was seen under the microscope which enabled Dr. Brown and Mr. Williams to detect a single particle of exhausted ginger in the presence of a much larger quantity of unexhausted ginger. Hence he had written to the wholesale dealers for a sample of the ginger which had been in question, and they had very courteously sent him a sample, which they stated to be Cochin ginger, and the same quality as that condemned by Mr. Blunt. He had also applied to one of the largest extractors in the country for specimens of ginger before and after exhaustion. He had not only analysed these, but had examined them most carefully with the microscope, but neither he nor several members of his staff, who were skilled observers, and in whose observations he had every confidence, could succeed in detecting the differences seen by Dr. Campbell Brown and Mr. Collingwood Williams. They could detect no difference at all between the starch in the exhausted and the unexhausted ginger, even knowing what the samples were and what to look for.

The following were the analytical data yielded by the samples in question :

	Total Ash. Per cent.	Soluble Ash. Per cent.	Cold-water Extract. Per cent.
<i>Newport Sample :</i>			
Allen	2.65	1.30	6.20
Blunt	2.74	1.24	6.20
<i>Cochin Ginger, from wholesale dealers (said to be the same as the Newport sample)...</i>	4.62	2.64	10.20
<i>Gingers from Wholesale Extractors :</i>			
Original	—	2.45	14.80
Exhausted	—	0.95	4.80

Mr. Allen added that he regarded the soluble ash and cold-water extract as among the most useful data in examining gingers, as it was evident that they would be materially diminished by any process of maceration, whereas such treatment would not necessarily affect the essential oil and resin. He (Mr. Allen) might remind the meeting that the figures for resin and essential oil in ginger, obtained by Dr. Dyer, were found to vary within very wide limits, so much so that he (Mr. Allen) had long since ceased to regard the determinations as having any practical value. On the other hand, he thought Mr. Stock's practice of determining the potash in ginger was valuable.

As a result of his experiments and inquiries, he was satisfied that the Newport sample contained a notable proportion of exhausted ginger, and was strongly of opinion the microscopic appearance of the starch was valueless as a means of detecting exhausted ginger.

Dr. DYER said that he had always extracted first with ether, and then with alcohol. If the extraction were made with alcohol only, the same result was not obtained. He supposed that "resin" meant alcoholic extract pure and simple.

Some time subsequently to the paper which had been read before the Society on the subject of ginger by himself and Mr. Gilbard, Mr. Allen had read a paper, and, in the course of the discussion on that paper, he (Dr. Dyer—see ANALYST, vol. xix., p. 127) gave some analyses of Japan ginger. Mr. Chattaway on that occasion pointed out that it was not at that time, practically speaking, a commercial article. He himself did not think it was, and believed that it was not imported now to any great extent; but some of it came into the market, and as it was of very low quality, the defence raised in several of these ginger cases was that the ginger was Japan ginger, and that Japan ginger gave a low percentage of soluble ash, etc.

He had quite recently had before him a sample of ground Japan ginger containing only 0.9 per cent. of soluble ash, and reported his opinion that it consisted of partially exhausted ginger. The grinder, however, had bought the ginger as imported, and ground it, and, as far as he knew, it had not been exhausted. It had, however, been "washed"—but not by the grinder himself, who had given it out for that purpose. The spice-grinder was not necessarily the washer; there were people who prepared ginger, taking the root as imported, cleansing and lime-washing it, and dressing it up for the market. As far as he (Dr. Dyer) could make out, this was a special trade.

The matter was, of course, a very serious one. To investigate it, he obtained samples of Japan ginger before and after being sent to the "washer," the latter being still in the whole or unground state. The results of the analysis were :

	Japan Ginger as imported. Per cent.	Japan Ginger after washing. Per cent.
Total ash	4.4	3.8
Soluble ash	1.6	1.3
Cold-water extract	12.6	9.2
Ash of cold-water extract	2.6	2.0
Approximate volatile oil7	.5
Fixed ether extract	4.4	4.3
Alcoholic extract after ether extraction	5.6	3.7

There was, as would be seen, a very distinct difference. The first sample to which he had referred, which had not been "exhausted" as far as the grinder was aware, contained, as he had said, only 0.9 per cent. of soluble ash, and the other factors were lower right through. It was difficult to say exactly how far such exhaustion was due to carelessness in washing, or how far it was a matter of deliberate intent, as suggested by the President. But, at all events, a considerable degree of exhaustion had taken place, the wholesale spice-dealer thinking all the while that he was practically grinding the stuff as imported, except that it was cleaner. He had never met, even in "washed" samples, with such low figures except in Japan gingers; but he was not at all satisfied that these Japan gingers were altogether genuine when they were imported, and he thought that some of the spice people themselves were not satisfied on the point either.

Mr. CHATTAWAY inquired whether the ginger referred to by Dr. Dyer was washed in this country.

Dr. DYER replied that it was; but whether it had been also treated before leaving Japan he did not know.

Mr. HEHNER said that in any case, whatever opinions might be held as to the treatment the sample under dispute had undergone, it was one of an exceedingly doubtful character, and, in order to rehabilitate it, it had to be compared with samples like the Japan ginger to which Dr. Dyer had referred, but which, coming from a different country, had absolutely no connection with this sample at all. It was interesting, no doubt, to find such samples of ginger, possibly from not very highly-cultivated plants; but in defending commercial samples upon the strength of abnormal ones, were they not in a somewhat similar position to that of an analyst who went into court to support a vendor of milk containing 7.5 per cent. of solids-not-fat?

With regard to the supposed change in the condition of the starch granules, he might remark that almost every starch that was sold in commerce had been washed either with water or with alkali, and yet the starch granules remained intact—a fact which was absolutely at variance with the theory as to the bursting of the starch granules in ginger by washing alone, which had been referred to. It was not to be imagined that ginger starch behaved differently in this respect to other starch granules.

Dr. J. A. VOELCKER said it seemed that an analyst was expected to be able to

distinguish between "washed" and "extracted" ginger. From the analytical figures before them, it would appear that washing effected very much the same thing as "extraction." Was the washed ginger, so to speak, to be passed as "genuine"? or was it an intermediate stage between genuine and exhausted?

Dr. DYER said that in the paper he had referred to—by himself and Mr. Gilbard—some analyses were given of washed varieties of several kinds of gingers, which all came from a very good spice house. These were evidently properly washed, and not exhausted in the process of washing; the figures obtained from those samples, although washed, were not those of exhausted ginger, like the others he had mentioned. Washing was not supposed to involve exhaustion; but there was a great difference between merely dipping the ginger into water and leaving it to soak, perhaps for days, either with or without intent to utilize the extract.

Mr. F. HUDSON-COX said that he had obtained the following figures from two samples of unexhausted (Cochin) and exhausted (Jamaica) ginger, which he had procured respectively from Messrs. Wright, Layman and Umney, and from another firm:

				Unexhausted.			Exhausted.
Total ash	6.06	4.42
Soluble ash	2.64	1.64
Insoluble ash	3.42	2.78
Cold-water extract	12.60	13.00

With the exhausted sample he had made several slides, to try and find burst starch granules, and, with the aid of a little imagination, he did succeed in finding one or two in about half a dozen slides, but no more.

Dr. DYER said he had come to the conclusion that a more constant and a nicer figure to work with than the soluble ash was the ash of the cold-water extract. It was a larger figure. The cold-water extract was merely dried and incinerated. In the determinations of soluble ash, variations were apt to be caused by different methods of burning or extracting. It must be remembered, however, that the figure from the ash of the water extract was quite a different one from that of the ordinary soluble ash and was not directly comparable with it.

ON THE DETERMINATION OF STEARIC ACID IN FATS.

By OTTO HEHNER AND C. A. MITCHELL, B.A.

(Read at the Meeting, November 4, 1896.)

THE quantitative determination of the individual members composing a mixture of the fatty acids obtainable from any fat or oil is one of the most difficult problems of analytical chemistry, and one which is very far from being solved at the present time. Apart from the determination of certain constants, such as the iodine absorption, amount of potassium hydroxide neutralized, etc., little more can be done than a rough separation of the saturated from the unsaturated fatty acids. A quantitative separation of the unsaturated fatty acids has barely been attempted, if we except the determination of the amount of the hexabromide obtainable from the mixed fatty

acids of linseed and other drying oils, which measures the linolenic acid, to which one of us referred at a recent meeting of the Society of Public Analysts, and concerning which we hope to publish the results of our joint work at an early date. The few figures published by Hazura, based upon the proportions of hydroxyacids obtained by the action of potassium permanganate upon unsaturated fatty acids, though of high scientific interest, are obviously very rough and of no practical value.

For the separation of the solid fatty acids no quantitative method of any kind exists, and here, again, inferences based upon the determination of physical constants and of the equivalents of mixtures have been the only available means. If it be known that a mixture contains two saturated fatty acids only, and no more, the determination of the melting-point, or of the molecular equivalent, is a satisfactory way of arriving at their relative proportions. But, unfortunately, there is no practicable method of proving that two, and only two, acids are present; hence all determinations hitherto made must be accepted with a considerable amount of doubt.

Heintz's well-known method supplies the only means of obtaining an insight into the qualitative composition of a mixture of non-volatile fatty acids; and Liebig's analogous method affords a means of examining a mixture of volatile fatty acids. Heintz's method is, however, solely a qualitative one, and appears unsuited for analytical purposes. Without frequent repetition of the fractional precipitation, nothing like a perfect separation of two closely allied fatty acids can be obtained. As, however, we could find but few data on this point in literature, we thought it advisable to ascertain to what extent Heintz's method was capable of concentrating the fatty acid of higher molecular weight from a mixture of known composition by a single precipitation only.

FRACTIONAL PRECIPITATION.

Stearic Acid.—A considerable quantity of fatty acids obtained in the usual manner from cocoa-butter was dissolved in as little hot alcohol as possible, and, when cold, the crystallized mass was pressed in a linen cloth. The residue was again dissolved in hot alcohol and pressed. After repeating this crystallization ten times, the melting-point remained constant at 68.5°C . (Heintz gives 69°C . as the melting-point of stearic acid), and had a molecular weight of 283, as determined by the amount of KOH consumed and the percentage of barium in the barium salt (theory for stearic acid, 284).

Palmitic Acid.—In most of our experiments we made use of pure palmitic acid obtained from palm-oil, and prepared by Hopkin and Williams. This melted at 61.8°C . (Heintz, 62°C .), and had a molecular weight of 255.5 (theory, 256). Subsequently we prepared palmitic acid from Japan wax. To the experiments made with this substance we shall refer later on.

Precipitation with Barium Acetate.—A solution of barium acetate was prepared containing 8.94 grammes of barium acetate in 100 c.c. of water, of which solution 5 c.c. contained 0.241 gramme Ba, capable of combining with 1 gramme of stearic acid. The mixed fatty acids were dissolved in 100 c.c. of hot (methylated) alcohol of 98 per cent., and the required quantity of barium acetate solution was added drop by drop from a burette to the hot alcoholic fatty acid solution, which was shaken

after the addition of each drop. After cooling, the precipitates were filtered off, thoroughly washed with cold alcohol of 98 per cent., dried in the water-bath, and the barium contained in them determined by ignition with sulphuric acid. From the amount of barium sulphate obtained the molecular equivalent of the precipitated fatty acids was calculated, and from this the amount of stearic acid by the formula :

$$x = \frac{284(m - 256)}{m(284 - 256)}$$

where x is the required amount of stearic acid, m the molecular equivalent found, and 284 and 256 the equivalents of stearic and palmitic acids respectively.

The filtrate and washings from the first precipitate were mixed, and a second fraction of the fatty acids remaining in solution precipitated with barium acetate solution, and treated as before. In some cases a third and fourth precipitation was made. The results are given in the following table :

I.

Stearic Acid, gm.	Palmitic Acid, gm.	Barium Acetate Solution added.	Barium Salt, gm.	Containing Barium, gm.	Mol. Weight of Fatty Acids.	Stearic Acid, p.c. in ppte.	Weight of Recovered Stearic Acid, gm.
1	1	5 c.c.	(1) 0.9614	0.1919	275.5	71.7	0.549
		5 c.c.	(2) 0.4326	0.09	261.75	22.2	0.076
		5 c.c.	(3) Too small for accurate estimation.				0.625
1	2	5 c.c.	(1) 1.0046	0.2021	272.9	62.8	0.5039
		5 c.c.	(2) 0.9896	0.1985	273	63	0.4983
		5 c.c.	(3) 0.5122	0.1066	261.6	22.3	0.0904
							1.0926
1	3	5 c.c.	(1) 0.8830	0.1827	263.2	27.9	0.1953
		5 c.c.	(2) 0.9100	0.1863	267.3	42.8	0.3097
							0.5050
1	3	5 c.c.	(1) 0.8920	0.1842	264.4	30.9	0.2187
		5 c.c.	(2) 1.0315	0.2125	265.2	35	0.2866
							0.5053
0.5	0.5	2.5 c.c.	(1) 0.5376	0.1082	272.8	62.7	0.2692
1	1	2.5 c.c.	(1) 0.5246	0.107	268.3	46.6	0.1946
			(2) 0.5262	0.1065	270.4	54	0.2266
							0.4212

As a further means of getting some idea as to the composition of the precipitated fractions, we recovered the fatty acids by shaking the barium salts with ether and hydrochloric acid in a separatory funnel, washing the ethereal solution several times with water, evaporating the ether, and determining the melting-point of the mixed

fatty acids. We thought it well to check, on this occasion, the table of melting-points constructed by Heintz, referring to mixtures of stearic and palmitic acids. The following are our comparative results :

II.

MELTING-POINTS OF MIXTURES OF STEARIC AND PALMITIC ACIDS.

Stearic Acid, per cent.	Palmitic Acid, per cent.	Heintz* Melting- point, ° C.	Hehner and Mitchell, ° C.
100	0	69.2	68.5
90	10	67.2	66.5
80	20	65.3	64.2
70	30	62.9	61.5
60	40	60.3	59.4
50	50	56.6	55.6
40	60	56.3	55.5
32.5	67.5	55.2	54.5
30	70	55.1	54.2
20	80	57.5	56.5
10	90	60.1	59.0
0	100	62.0	61.8

It will at once be seen by a glance at these tables that any judgment as to the composition of a mixture of stearic and palmitic acids based on the melting-point can only be very roughly approximate, especially when the acids are present in about equal proportions, there being only about 1.5° C. difference between mixtures containing 50 and 30 per cent. of stearic acid. Still, it enabled us to some extent to confirm the results given in the first table, obtained by determining the equivalents of the mixed acids. In the following table the results given are based on the melting-points only.

III.

Stearic Acid, grms.	Palmitic Acid, grms.	Barium Acetate Solution added.	Melting-point, ° C.	Approximate Percentage of Stearic Acid in ppte.
0.5	0.5	(1) 2.5 c.c.	60.8	between 60 and 70
		(2) "	54.6	about 30
1	1	(1) 2.5 c.c.	59.4	60
		(2) "	59	nearly 60
		(3) "	58.6	between 50 and 60
		(4) "	54.2	30
0.5	1	(1) 2.5 c.c.	57	about 55
		(2) "	55.8	about 50
		(3) "	54.2	30
2.1	0.9	(1) 2.5 c.c.	64.0	nearly 80
		(2) "	65.0	between 80 and 90
		(3) "	65.8	" "
		(4) "	60.5	between 60 and 70
		(5) "	54.6	between 30 and 40

It is obvious from these results that by means of an aqueous solution of barium acetate, used in the proportion stated, but a very moderate concentration of the

* *Ann. Chem. Pharm.*, xcii., 295.

higher fatty acid can be accomplished in one precipitation. Curiously enough, it not infrequently happens that the second fraction contains a larger percentage of the higher fatty acid than the first portion. Quantitative results cannot possibly be obtained.

Precipitation with Lead Acetate.—As it might be thought that, owing to the employment of an aqueous solution, and the consequent somewhat sudden separation of the precipitate, a less perfect fractionation would take place than if the precipitate were allowed to form very slowly, we repeated the fractionation with the employment of an alcoholic solution of lead acetate. A solution, containing 6.67 grammes of the salt in 100 c.c. of alcohol, of which 10 c.c. were capable of completely precipitating 1 gramme of stearic acid, was made, and the required amount of this solution was added slowly to the hot alcoholic solution of the mixed fatty acids. The fraction crystallizing out on cooling was treated as described in reference to the preceding table.

IV.

Stearic Acid, gm.	Palmitic Acid, gm.	Lead Acetate Solution added.	Melting-point of Liberated Acids, °C.	Approximate Percentage of Stearic Acid.
0.5	1	(1) 5 c.c.	60.2	between 60 and 70
		(2) "	55	between 30 and 40
		(3) "	55.4	" "

Here, again, the separation was as incomplete as in Tables I. and III.

Precipitation with Magnesium Acetate.—A solution containing 18.835 grammes of magnesium acetate in 500 c.c., of which 10 c.c. were required to precipitate 1 gramme of stearic acid, was made and used in the following experiment:

Stearic Acid, gm.	Palmitic Acid, gm.	Magnesium Acetate Solution added.	Melting-point, °C.	Approximate Percentage of Stearic Acid.
0.5	0.5	5 c.c.	62	About 70

By these results we were confirmed in our opinion, that, while by oft-repeated fractionation a mixture of fatty acids may be made to yield the highest fatty acid in a comparatively pure condition, by a single precipitation no sharp separation can even approximately be obtained, however slowly the separation be allowed to take place. When more than two fatty acids are present in a mixture, the results must be still more doubtful. For quantitative analytical work, for which, of course, the method was never intended, it is not applicable.

After numerous attempts to separate the liquid and the solid fatty acids from each other by means of glacial acetic acid, which readily dissolves oleic, but at the same time not inconsiderable amounts of palmitic and of stearic acids, and by mixtures of acetic acid, alcohol, and water (after the method of David, *Compt. Rend.*, 86, 1416), we came to the conclusion that nothing like a sharp separation could be effected in this way. Somewhat better were the results obtained by the use of dilute alcohol alone. On grinding up the fatty acid obtained by the saponification of a solid fat, and decomposition of the resulting soap, with dilute alcohol (specific gravity 0.911)

in a mortar, we found that the unsaturated fatty acids were completely dissolved, the palmitic acid partially so, while the stearic acid remained almost completely undissolved. From 10 to 20 grammes of various fatty acid mixtures were thus ground up with 100 c.c. of the dilute alcohol, the latter removed by a filter-pump, and the residue was washed with 100 c.c. of dilute alcohol of the same strength. After pressing the residue in a filter-cloth, placed between blotting-paper, a pearly-white mass was obtained, which, after drying, showed an iodine absorption of from 2 to 3 per cent. From the molecular weight, ascertained by means of alcoholic alkali, the proportions of palmitic and stearic acids were calculated, on the assumption that nothing but stearic and palmitic acids were present—an assumption which could not be very incorrect, because the lower fatty acids are soluble to a considerable extent in alcohol of the strength mentioned.

VI.

	Fatty Acid, grms.	Washed Residue, grms.	Molecular Weight.	Stearic Acid Percentage in Washed Acids.	Stearic Acid Percentage in Original Acids.	Iodine Number of Original Fatty Acids.	Melting-point of ditto, ° C.
Butter (Brittany)	10	2.0	260.8	15	3		
Same sample ...			259.3				
			260.3				
Butter (Danish) ...	20	2.5	263.6	29	3.6		
			263.8				
" " ...	10	1.85	258	7	1.29		
Oleomargarine ...	10	4.0	267.3	42.8	17.12	46.5	43.2
Margarine ...	10	3.7	266.2	40	14.8	43.23	38
" " ...	10	4.0	265	34.4	13.76		(Dalcian.)
Mutton tallow ...	10	5.5	264	30	16.5	39.5	43.8
" " ...	20	11.3	265.8	37.4	19.47	35.6	47.8
Lard ...	20	7.5	258.8	18.6	3.2		
" " ...	20	7.0	260	15	5.2		

Although, when working under exactly the same conditions, fairly constant results could be obtained, the method effected at best only a concentration of the stearic acid, and afforded no certainty that the final product consisted of only two fatty acids, from the equivalent of which the proportion might be calculated. Although we worked as nearly as possible at a uniform temperature, variations could not be avoided, and this led us to abandon the search in this direction.

The following reasoning gave much greater hope of success: Imagine a mixture of fatty acids to be treated with an amount of solvent sufficient to dissolve the whole of the various components of the mixture. If the solvent were already saturated with one of the constituents of the fatty acid mixture to be examined, it is to be expected that the totality of the corresponding fatty acid contained in the mixture would remain undissolved. Or, if a mixture were heated with a solvent saturated at a certain temperature with one of the constituents sought to be determined, then, on cooling down to the corresponding temperature, the whole of the particular constituent might be expected to crystallize out quantitatively, provided that the other

constituents of the fatty acid mixture exercised no restraining influence by adding to the solubility.

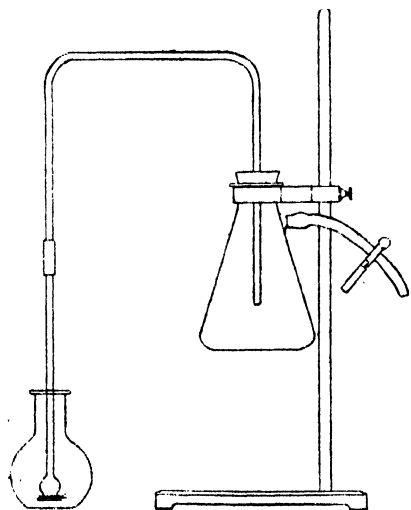
In principle this is the method of Casamajor for the quantitative separation of cane from invert sugar. Casamajor washed the sugar to be examined with methyl-alcohol saturated with cane-sugar.

We chose the freezing-point of water as the most suitable constant temperature, it being easily maintained, and the solubility of stearic and the other acids occurring in fats being suitable for accurate work.

After some preliminary experiments with ether and other solvents, which we had to reject as taking up too much stearic acid, we finally fixed upon alcohol (methylated) of a specific gravity of 0.8183, containing 94.4 per cent. of alcohol by volume. It will be gathered from what follows that the exact strength of alcohol is a matter of no consequence; but the solubility determinations given here apply only to alcohol of that strength.

For maintaining a constant temperature we employed an ice-chest, constructed of a metal box, to the interior sides of which were soldered suitable sockets to receive the clamps necessary to hold the small flasks, submerged up to the neck in ice-water, in which the determinations were carried out. The metal box was fitted in a wooden box, and the space between the metal and wood was packed with wool and sawdust, while a cushion of wool and flannel was placed between the lids of the metal and wooden boxes.

For the preparation of the stearic solution about 3 grammes of pure stearic acid were dissolved in about a litre of warm alcohol of specific gravity 0.8183, and the stoppered bottle containing the solution placed overnight in the ice-water (which contained lumps of ice) in the chest, so that the bottle was submerged up to the neck. After twelve hours a considerable portion of the stearic acid had crystallized out. The saturated mother-liquor was syphoned off without removing the bottle from the ice-water. The filtering syphon consisted of a small thistle funnel twice bent at right angles, fitting with its straight limb into a flask in connection with a suction-pump. The bulb of the funnel, which was submerged in the ice-cold solution, was covered over with a piece of fine calico. On applying suction, a perfectly clear stearic solution was obtained, saturated at 0° C., or, rather, at 0.2° C., which was the temperature almost constantly shown by a standard thermometer.



A precisely similar mode of filtration was also adopted in the quantitative experiments on mixed fatty acids, the thistle funnel used being a miniature one, with a bulb not larger than about $\frac{1}{4}$ inch in diameter. The accompanying drawing will sufficiently explain the apparatus.

VII.

Solubility of Stearic Acid in Alcohol of Specific Gravity 0.8183 at 0° C.

Stearic Acid taken, gram.	Alcohol, c.c.	Time of Cooling, hours.	Residue obtained by evaporating 10 c.c. of the Filtered Solution, gram.
0.2	100	12	0.0155
"	"	36	0.0153
"	"	84	0.0153
0.5	100	12	0.0158
"	"	36	0.0145
"	"	84	0.0140
0.3	100	48	0.0144
"	"	84	0.0142
0.3	100	12	0.0157
"	"	36	0.0149
0.4	100	12	0.0158
"	"	36	0.0148

VIII.

Solubility of Palmitic Acid in Alcohol of Specific Gravity 0.8183 at 0° C.

Palmitic Acid taken, grams.	Alcohol, c.c.	Time of Cooling, hours.	Residue obtained by evaporating 10 c.c. of the Filtered Solution, gram.
2	100	12	0.1298
		36	0.1244
		60	0.1211
		84	0.1134
		108	0.1086
		132	0.1044
		156	0.1028
2	100	12	0.1320

From the two preceding tables it appears that it is easy to obtain constant results with stearic acid, and that supersaturation does not readily occur. The solubility of palmitic acid in alcohol of the same strength as that employed for stearic acid is about eight times greater than that of stearic acid, and constant results are far less easy to obtain.

Experiments with known Amounts of pure Stearic Acid.—Accurately weighed quantities of pure stearic acid were placed in weighed flasks, provided with a rubber cork, capable of holding about 150 c.c. From 50 to 100 c.c. of the saturated stearic solution were added, the flask closed, and the contents gently warmed on the water-bath till all the stearic acid had dissolved. The flask was then clamped in the ice-chest, and kept in ice-water overnight. In the morning the contents of the flasks

were, as a rule, gently shaken, without being withdrawn from the ice-water, in order to promote crystallization, and again left for at least half an hour. The alcohol was then filtered off by means of the arrangement described, care being taken to draw off the solution as completely as possible. The residue was washed three times successively with 10 c.c. of the saturated stearic solution cooled down to 0° C. The small funnel was then detached from the suction arrangement, and washed with hot alcohol, the washings falling into the flask which contained the residue, the flask having been previously taken out of the ice-chest. The alcohol was then evaporated off, and the residue dried at 100° C. to constant weight.

Since the sides of the interior of the flask, as well as the residue of crystallized stearic acid, retained a small amount of the stearic alcohol solution, a correction, experimentally found to amount to 0.005 gramme, had to be applied, this amount being deducted from the total weight found. In almost all cases the precipitated stearic acid formed a small compact mass after filtration, which retained a minute amount only of the washing solution.

The following results were obtained :

Stearic Acid taken, gram.	Saturated Alcohol, c.c.	Total Residue found, gram.	Stearic Acid, after deducting 0.005 gramme, gram.
0.0188	100	0.021	0.016
0.0220	100	0.025	0.020
0.0320	100	0.0381	0.0331
0.0381	50	0.0445	0.0392
0.1072	50	0.113	0.108
0.1356	100	0.1414	0.1364
0.1414	100	0.1440	0.1390
0.2137	100	0.2206	0.2156
0.2282	100	0.2334	0.2281

As a check, we also made a number of experiments by dissolving weighed quantities of stearic acid in the saturated alcohol, leaving the flask overnight in ice, and, instead of weighing the deposit, evaporated measured quantities of the filtrate, thus determining the amount of stearic acid in solution. If this corresponded with the amount previously found to be soluble in 10 c.c. of alcohol, it followed that the whole of the added stearic acid had separated.

X.

Stearic Acid, gram.	Saturated Alcohol, c.c.	Residue in 10 c.c. after 12 hours, gram.
0.1955	100	0.0155
0.5090	100	0.0150
0.3130	100	0.0157
0.4010	100	0.0158

It will be seen that the amount left in solution perfectly corresponded with that contained in the stearic alcohol solution, and that therefore none of the added stearic acid could have remained in solution.

Having thus satisfied ourselves that pure stearic acid could be determined with

a satisfactory degree of accuracy, it remained to be seen whether, in the presence of other fatty acids, equally satisfactory results could be obtained, or whether other fatty acids would themselves exercise a solvent action upon the stearic acid.

XI.

Influence of lower saturated Fatty Acids.

Stearic Acid taken, grm.	Lower Fatty Acid employed.	Stearic Acid found, after deducting 0·005 gramme, grm.
0·0556	5 c.c. butyric acid.	0·0582
0·0476	5 c.c. acetic acid.	0·0468
0·0990	1·5 gramme cocoa-nut fatty acids.	0·1070
0·1060	0·9079 gramme " "	0·118
0·2020	0·5130 " "	0·220

XII.

Influence of Palmitic Acid.

Stearic Acid taken, grm.	Palmitic Acid added, grm.	Stearic Acid found, after deducting 0·005 gramme, grm.
0·2026	0·5472	0·2032
0·1016	0·5354	0·1074
0·0520	0·600	0·055

Influence of unsaturated Fatty Acids.—The oleic acid used for the following experiment was commercial oleic acid, and was not free from stearic acid, 3 grammes, crystallized in the manner described from stearic alcohol, giving 0·026 gramme of stearic acid. A corresponding deduction had therefore to be made in the following experiment:

XIII.

Stearic Acid, grm.	Crude Oleic Acid, grm.	Stearic Acid found, grm.
0·2997	3·200	0·3355

Deducting from the latter figure 0·0278 gramme, the amount of stearic acid obtainable from the 3·2 grammes of oleic acid, and 0·005 gramme for correction, there remain 0·3027 gramme of stearic acid recovered from the 0·2997 gramme taken, or 1 per cent. too much.

Influence of saturated and unsaturated Fatty Acids together.—Fatty acids prepared from hog's lard were first crystallized by themselves, and afterwards with different quantities of added stearic acid, with the following results:

XIV.

Lard Acids taken, grm.	Stearic Acid added, grm.	Stearic Acid obtained after deducting 0·005 grm., grm.	Added Stearic Acid recovered, grm.
0·5	none	0·0242	—
0·5	0·050	0·0738	0·0496
0·5	0·080	0·1058	0·0816

It is thus shown that stearic acid, added to a mixture of fatty acids, can be completely recovered in the manner described.

Experiments with commercial Stearic Acid.—The sample employed had an iodine absorption of 2·3 per cent., and melted at 56·2°.

XV.

Commercial Acid taken, gm.	Stearic Alcohol added, c.c.	Amount of Deposit, - 0·005 gm., gm.	Per cent. of Stearic Acid found.
0·1042	50	0·0490	47·02
0·1480	50	0·0702	48·7
0·2510	50	0·1223	48·7
0·2900	50	0·1468	50·6
0·3984	100	0·1932	48·5

XVI

Experiments with Commercial Lards.

Lard, gm.	Iodine No.	Stearic Acid, - 0·005 gm., gm.	Stearic Acid, per cent. in Fatty Acids.
1. 0·9794	61·2	0·1338	13·06
2. 0·5065	not taken	0·0635	12·53
0·8328		0·1090	13·08
1·0604		0·1359	12·90
3. 0·4970	57·5	0·0801	16·11
1·0178		0·1634	16·05
4. 1·0987	61·2	0·0669	6·09
0·9990		0·0764	7·6
1·2878		0·0782	6·07
5. 0·5150	65·66	0·0548	10·64
1·0410		0·1030	9·89
6. 0·5000	63·58	0·0372	7·4

Determination of Stearic Acid in Fat from different Parts of the same Pig.—It is well known that the proportion of the liquid and solid fat varies very considerably in the fat taken from different parts of the same animal. We thought it would be of interest to ascertain whether, in this case, the alteration affected only the proportions of fluid (unsaturated) and solid fatty acids, or whether the latter themselves varied in composition. The animal from which the fat was taken was a Somersetshire pig, six months old. The following table contains the results:

XVII.

Part of Pig.	Fatty Acids taken, gm.	Melting at.	Iodine Absorption.	Stearic Acid, - 0·005 gm., gm.	Melting-point of Deposit.	Stearic Acid per cent.
Head	0·5212	34·8° C.	67·7	0·0452	67·8° C.	8·67
	1·0481			0·0993	66·2	9·47
Ham	0·5006	34·6	61·6	0·0438	67·5	8·74
	1·0044			0·0906	67·3	9·02
Breast	0·5152	36·8	64·2	0·0610	67·2	11·84
	1·0219			0·1114	66·8	10·9
Flare	0·4936	40	52·8	0·0774	66·5	15·7
	1·0096			0·1450	67·2	14·4
Back	0·5002	35·6	67·9	0·0430	67	8·59
	0·9998			0·0910	66·5	9·2

Flare from another pig :

1.5221	0.1940	67.2	12.7
1.1320	0.1386	67.0	12.24

It will be observed that the melting-points of the deposits obtained were in all cases slightly lower than that of pure stearic acid, but a mere trace of fluid fatty acid would be fully sufficient to account for the depression.

Assuming that the iodine number measures only oleic acid, and that 100 parts of the latter absorb 90 parts of iodine, it is easy to calculate the percentage of stearic acid in the saturated fatty acids contained in the total fatty acids from the fat of the animal referred to. On making this calculation, it is found that the following are the percentages of stearic acid in the saturated fatty acids :

Head	35	per cent.
Ham	28.1	"
Breast	39.6	"
Flare	36.4	"
Back	36.1	"

It follows that, in the case of this particular animal, at least, the variation in the consistency of the fat from various parts was almost entirely due to fluctuations in the amount of oleic acid.

Stearic Acid in Fat from different Parts of the same Sheep.—The different specimens of fat were taken from a Scotch sheep eighteen months old :

XVIII.

Part.	Fatty Acids taken, grm.	Iodine No.	M.P. of Fatty Acids.	Stearic Acid, 0.005 gramme, grm.	Percentage of Stearic Acid.
Back	0.5280	61.3	41.4	0.1310	24.8
Neck	0.5324	48.6	42.2	0.0876	16.4
Breast	0.7146	58.2	33.8	very slight	about 1
Ham	0.5100	50.6	40.8	no deposit after two days	
Kidney	0.5030	48.16	45.6	0.1318	26.2
"	1.0175	—	—	0.2820	27.7

Calculating, as before, the percentage of stearic acid in the saturated fatty acids, we find :

Back	78	per cent.
Neck	36	"
Breast	3	"
Ham	none	
Kidney	58	per cent.

The ham-fat was fluid at ordinary temperature, and that from the breast almost fluid.

The following results may find a place here, as they afford an indirect corroboration of the correctness of the stearic acid determinations : A sample of sheep's kidney fat-acids, having an iodine absorption of 48.16 and a molecular equivalent of 276.1 (as determined by the amount of KOH consumed), was found to include 26.2 per cent. of stearic acid. The iodine number corresponds to 56.0 per cent. of oleic

acid, assuming that the whole of the unsaturated acid is oleic. Hence the amount of palmitic acid would be, by difference, 17·8 per cent, assuming that the fatty acids consisted of stearic, palmitic, and oleic only. Calculating the total molecular equivalent from these data, the following figures are obtained :

Oleic	$0\cdot56 \times 282 = 157\cdot92$
Stearic	$0\cdot262 \times 284 = 74\cdot41$
Palmitic	$0\cdot178 \times 256 = 45\cdot57$

Total ... 277·90

against 276·1 found by direct titration.

XIX.

Determination of Stearic Acid in Miscellaneous Fats.

	Taken, grm.	Iodine No.	Stearic Acid, - 0·005. grm.	Percentage in Fatty Acids.
Beef-stearin ...	0·3024	2·0	0·1516	50·19
" ...	0·4174		0·2131	51·05
Oleomargarin ...	1·0107	46·50	0·2295	22
" ...	0·5192		0·1104	21·26
" ...	1·1100		0·2630	23·6
Margarin—I. ...	1·0035		0·2495	24·8
" —II. ...	0·5000	41·19	0·0586	11·72
Horse kidney fat ...	0·701	85·4	no deposit	
Cotton-oil " stearin " ...	0·9945		0·0334	3·3
Stillingia tallow ...	—	22·87	no deposit	
Cocoa-butter ...	1·0168		0·4064	39·9
" ...	0·9548		0·3878	40·6
Maize-oil ...	5·4186	122	no deposit	
Almond-oil ...	5·0236	95·68	no deposit	
Olive-oil ...	5·5558		no deposit	
Earthnut-oil ...	1·0648		0·0751 (M P 67° C.)	7

XX.

Examination of Lard Crystals separated from Ether.—It is well known, to those who are accustomed to test for beef-stearin in lard, that the form of the crystals obtained from pure lard is subject to considerable variation. While from some lards (the softer kinds) nothing but broad plates with pronounced chisel-shaped ends are obtained, from the harder kinds (flare lard) bunches of more pointed, though still chisel-edged, needles crystallize. When the crystals are repeatedly recrystallized from ether, they, as a rule, become more and more needle-like, until they are practically undistinguishable from beef-stearin crystals. One of us illustrated this behaviour some time ago, before the Society of Public Analysts, by the exhibition of a large number of photo-lantern slides. After finding the remarkable differences in the amounts of stearic acid contained in various parts of the fat of the same pig, stated above, we thought that determinations of stearic acid in lard crystals might throw light upon this subject. No one, as yet, has attempted to explain the cause of the difference between the shapes of lard crystals and of beef-stearin crystals; and it is not known whether the phenomenon is due to different isomerides, or simply to the proportion of stearic to palmitic or other acids contained in the deposits.

One hundred and forty grammes of a sample of flare lard, having an iodine absorption of 57·5, and the fatty acids of which contained 16·11 and 16·05 per cent. of stearin, were dissolved in a litre of warm ether. This is nearly the proportion of ether to lard generally employed in Stock's mode of working the Belfield test. Next morning there was an abundant deposit of crystals, showing the characteristic chisel-shaped ends. A small portion of the crystals separated by filtration was saponified, etc. The free fatty acids thus obtained were found to contain 32·4 per cent. of stearic acid. The ethereal solution poured off from the crop of crystals was evaporated, and part of the resulting residue decomposed to obtain the free fatty acids. These contained only 15 per cent. of stearic acid.

The main crop of crystals was then recrystallized from ether. The crystals were now needle-shaped, but had still distinct chisel-shaped ends. The fatty acids obtained from these contained 47·6 per cent. of stearic acid, while the fatty acids obtained as before from the second solution contained 17·23 per cent. stearic acid.

The crystals were crystallized a third time from ether. The percentage of stearic acid had now risen in the fatty acids from the crystals to 59 per cent., while the fat in solution contained 33·2 per cent. This third crystallization was hardly distinguishable in form from beef-stearin crystals.

It can hardly be doubted, from a consideration of these results, that the form of the beef crystals is solely due to a larger proportion of stearic acid than can be obtained from a pure lard by a single crystallization. When it is considered that pure lard contains, as far as our results go, not more than about 15 per cent. of stearic acid, while beef-stearin contains about 50 per cent., it is intelligible that from a beef-stiffened lard a deposit with a larger percentage of stearin is likely to be obtained than from lard alone. At the same time, the subject is quite worthy of a much more extended investigation.

The results also show that very many crystallizations would be required to separate the glycerides of palmitic and stearic acids fairly completely, as was pointed out by Heintz many years ago.

XXI.

Examination of Samples of Butter.—We have made considerably over 100 determinations of stearic acid in the fatty acids of butter, utilizing for this purpose the fatty acids remaining behind after analyses by the Reichert method. In a great many cases no deposit at all, or only a very minute quantity, separated from the stearic alcohol. In some cases, however, phenomena, apparently those of supersaturation, occurred. Occasionally, on examining the flasks after opening the ice-chest in the morning, the solution was perfectly clear, but after shaking the contents, and leaving them for some time longer in the ice, a small but increasing number of crystals formed. We have not quite concluded our investigation into the cause of this phenomenon, and as the question of the examination of butter, with a view to distinguish it from margarine mixtures, is of exceedingly great practical importance, we propose to deal with this part of the subject in a separate communication. Inasmuch as all constants which are now utilized for the discovery of admixtures with butter-fat are occasionally breaking down, it would be a matter of

obvious importance if a constituent which is never absent from butter substitutes, viz., stearic acid, could be shown to be absent from butter-fat, or to occur in it only in minute quantities. Analysts would then, for the first time, be in a position to distinguish absolutely between pure and adulterated butter. For the purpose of the investigation, we would thank those of our colleagues who take an interest in the subject to kindly furnish us with small samples of undoubtedly genuine butters, normal and abnormal, together with their results of analysis, including any or all of the following: Reichert number, insoluble fatty acids, refractive index, together with any details that may be of interest or importance.

XXII.

On the Abnormal Behaviour of Palmitic Acid obtained from Japan Wax.—Japan wax, which consists, or is believed to consist, mainly of palmitin and free palmitic acid, is considered to be a ready source of palmitic acid. We prepared a quantity of the acid, possessing apparently all properties of palmitic acid, from Japan wax. On making mixtures of this acid with known amounts of stearic acid, we were surprised to find that only a part, or none at all, of the added stearic acid could be obtained by crystallization from the stearic alcohol, as the following figures will show:

Stearic Acid. gram.	Japan-wax Acid. gram.	Time of Standing.	Stearic Acid found. gram.
0.1410	0.5	8 days	0.0438
0.2500	0.5	12 hours	0.1542
0.0200	0.5	12 hours	turbidity only
0.2000	0.5	60 hours	0.1800
0.4000	0.5	12 hours	0.3800
0.2370	0.5	4 days	0.2217
0.1500	0.5	4 days	turbidity only
0.1000	0.100	12 hours	very slight precipitate
0.0500	0.1	3 days	" "
0.05	0.2	3 days	" "
0.025	0.225	3 days	" "
None	0.5	2 days	none

Without extended investigation, it is impossible even to guess at the reason of the abnormal behaviour of the acids of Japan wax. Whether they combine with the added stearic acid, or cause the latter to be etherified by the alcohol, we are unable to say. The whole subject of Japan wax is worthy of investigation, as the statements made concerning it are somewhat contradictory. In any case, the failure to apply our method to Japan-wax acids does not directly bear upon the question of the determination of stearic acid in ordinary fats and oils, in which we are quite satisfied that acid can be determined with reasonable accuracy.

The principle underlying the above method may be also applied to the determination of other fatty acids, such as palmitic and arachidic acids. In the former case the results would of necessity be less exact on account of the far more considerable solubility of palmitic acid and the greater correction number to be applied. For arachidic acid, on the other hand, the method ought to do good service, as it may safely be assumed that its solubility is far less than that of stearic acid. We hope to recur to this subject at some future time.

To summarize in a few words the method which we recommend : Prepare a supply of alcohol saturated at 0°C . with pure stearic acid, or with stearic acid which only contains traces of palmitic acid. Dissolve from 0.5 to 1 gramme of the mixture of the fatty acids to be examined, if these are solid, or about 5 grammes if fluid, in about 100 c.c. (exact measurement is not necessary) of the stearic alcohol solution. Leave in an ice-bath overnight, agitate the mixture next morning, and allow to stand in ice for a short time; filter off while the mixture remains in ice, wash with stearic alcohol at 0°C ., dry and weigh. Determine the melting-point of the product, which should not be much less than 68.5°C .

DISCUSSION.

Mr. ALLEN said that the subject was one upon which only limited information existed, and he congratulated the authors upon having made a very great advance. It was only in recent years that anything was possible in the way of quantitative differentiation of the fatty acids. Something could be done with the aid of the iodine absorption, but, as had been pointed out in the paper, this had to be taken as representing the proportions in which the saturated and unsaturated fatty acids were present. Now, however, the authors of the paper had made it possible to distinguish between the stearic acid and the other saturated fatty acids. The figures seemed quite conclusive that the method was a sound and practical one, and as good a step as could be hoped for towards the attainment of the actual determination of stearic acid as distinguished from other of the solid saturated fatty acids.

It was not possible to criticise the paper. All one could say was that it showed an enormous quantity of work. Only those who accumulated figures like these, and knew the number of disappointments to be experienced before they could arrive at anything like conclusions which they could themselves trust, or put before others, were aware of the enormous labour involved in researches of this kind. This was one of the many investigations with which the Society had been favoured by Mr. Hehner and his co-worker in this direction, and he (Mr. Allen) thought they were very much indebted to Mr. Hehner and his collaborator for placing these results before them. It constantly happened in the case of a new method of investigation (a striking example being afforded by the Röntgen rays), that there were directions, which did not occur to the original discoverer or inventor, in which the method could be usefully applied, and he was inclined to think that the process for determining stearic acid devised by the authors of the paper would be found in the future to be of very great assistance in the detailed examination of various kinds of fats.

Mr. HEHNER said that there were very many practical questions connected with this investigation which still required solution. He did not suggest that the method they had described was an ideal one; in fact, it was not a method which appealed to his enthusiasm at all. He would have much preferred a strictly chemical method to one based upon differences in solubility, and he would be the first to abandon the process described in the paper if a good chemical method were devised. He had himself made unsuccessful attempts in that direction. He thought, however, that they had proved the practicability of the method, the only exception being in

the case of Japan wax, which was not an edible fat, and which had only come into the investigation incidentally. With ordinary fats there was not the least difficulty, but the method required a strict adherence to details.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

ORGANIC ANALYSIS.

Estimation of Uric Acid in Guano. A. Stutzer and A. Karlowa. (*Chem. Zeit.*, 1896, xx., 721.)—One or two grammes of the finely-divided sample are weighed into a porcelain basin, covered with water and faintly acidified with hydrochloric acid. The latter is then completely evaporated off on the water-bath, 100 c.c. of water containing 3 grammes of piperazine in solution added, and the whole boiled for a minute. After filtration the liquid is thoroughly cooled, a little phenolphthalein introduced, and sufficient hydrochloric acid to remove the alkaline reaction. 10 c.c. of 10 per cent. hydrochloric acid are next added, the mixture well shaken (preferably by mechanical means) and allowed to stand for twelve hours. The deposit is collected on a filter-paper, and washed with 1 per cent. HCl till the filtrate measures exactly 200 c.c. The filter and its contents are subjected to a nitrogen estimation, and the result calculated into uric acid by means of the factor 2.994, an allowance of 3 milligrammes being finally made for the proportion dissolved in the 200 c.c. of washings. Two samples of Peruvian guano examined in this way gave 27.60 and 23.82 per cent. of uric acid respectively.

The authors have determined the solubility of uric acid in 1 per cent HCl under the above conditions, finding it to be in the case of the purest white powder of commerce 1 in 11,890 parts, but in the crystalline form 1 in 43,478 parts. As both preparations were perfectly pure, this difference must have been due solely to physical causes.

F. H. L.

A New Reaction for Asparagine. L. Moulin. (*Jour. Pharm. Chim.*, 1896, xvi., 543.)—Asparagine treated with sulphuric acid and a little resorcin behaves like saccharin. On warming, the liquid becomes yellowish green, and when diluted with water and saturated with ammonia or soda, gives a green fluorescence resembling that of resorcin itself. Crystals of asparagine purified by repeated recrystallization gave the reaction, as did also the crystalline product prepared from a cold extract of liquorice.

C. A. M.

New Reaction of Picric Acid. A. Swoboda. (*Zeits. österr. Apoth. Ver.*, 1896, xxxiv., 617; through *Chem. Zeit. Rep.*, 1896, 243.)—When solutions of picric acid and methylene blue are mixed together in the cold water, a flocculent violet precipitate is immediately formed, which is soluble in ether, chloroform, and hot water with a blue

or green colour. In the case of wooden toys, the methylene blue solution may be applied directly to the object, any varnish being previously removed by a few drops of alcohol. If a little chloroform is then added to dissolve the precipitate, and the solvent is evaporated off, the surface of the article will be found to be stained violet.

F. H. L.

A Modification of Goldenberg's Method for Tartar Estimation. M. Zecchin. (*Staz. Sper. Ag. Ital.*, xxviii, 788.)—Goldenberg's method (*ANALYST*, xxi., 76) is somewhat long, necessitating three filtrations and one evaporation; moreover, during the evaporation a small portion of the acid tartrate may be re-converted into normal tartrate.

The author proposes the following modification: Weigh out 3.75 grammes of the finely-powdered substance, and dissolve in hydrochloric acid on the water-bath; after 10 to 15 minutes make up to 100 c.c. and filter off 50 c.c. Add solid potassium carbonate in very slight excess, boil for some minutes, and make up to 100 c.c. Of this solution filter off 20 c.c., add 5 c.c. of glacial acetic acid and 100 c.c. of a mixture of equal parts alcohol and ether; shake well, and let the solution stand in a corked flask for 4 to 5 hours. Filter and wash the precipitate with alcohol and ether till neutral, and dissolve in boiling water; titrate with $\frac{N}{10}$ alkali, using phenolphthalein as indicator; each 1 c.c. indicates 2 per cent. of tartaric acid.

Six test analyses are given which agree well with the Goldenberg method. The author's method has the advantage of rapidity, as an analysis can be finished in a day.

H. D. R.

A Method for the Determination of the Diastatic Capacity of Malt. A. R. Ling. (*Jour. Fed. Inst. Brew.*, 1896, ii., 335-346.)—In determining the diastatic power of a malt by Lintner's method (*ANALYST*, this volume, p. 124) it frequently happens that a second series of tests is required, especially with distillers' malts, in which the point of reduction often lies between the first and second tubes. The author obviates this by titrating with a standard solution of glucose the unreduced Fehling's solution in those tubes in the first test in which reduction is almost complete, a concordant number of values being thus obtained. The glucose solution is conveniently prepared by dissolving 2 grammes of pure crystallized glucose (dextrose) in water and diluting to 1 litre. The copper contained in 10 c.c. of Fehling's solution should be entirely precipitated by 25 c.c. of this solution, but it is necessary to check this by titration, potassium ferro-cyanide being used as indicator.

In making a determination the contents of the selected tubes are transferred to a boiling flask before titrating, and it is essential that only those tubes should be taken in which the reduction is nearly complete. If those but slightly reduced are titrated, the results are altogether erroneous. In dealing with highly diastatic malts it is advisable to dilute the extract to half or quarter its strength.

The following example shows the accuracy of the method:

Two tubes containing 0.15 c.c. and 0.2 c.c. of malt-extract respectively required

5.5 c.c. and 3.8 c.c. of glucose solution (1 c.c. = 0.4 c.c. Fehling's), DP = 37.3 and 34.8. The results are calculated by the formula,

$$DP = \frac{10(5-y)}{5x},$$

in which

DP = the required diastatic capacity ;

x = the volume in c.c.'s of normal malt-extract taken ;

y = the volume in c.c.'s of un-reduced Fehling's solution found by titration.

C. A. M.

Notes on the Gravimetric Estimation of Sugar by Fehling's Solution. H. Elion. (*Rec. trav. Chim. des Pays-Bas*, 1896, xv., 116; through *Chem. Zeit. Rep.*, 1896, 243.)—In working this process the following points should be observed: (1) Treatment of the asbestos filter with alkali and nitric acid does not prevent it from constantly losing weight; the tube should therefore be weighed before the analysis, and again after the reduced copper has been dissolved, the mean value being taken as the true tare. (2) As some decomposition of the Fehling's solution is apt to occur during the boiling (*cf.* ANALYST, xx., 228), it is advisable to carry out a blank experiment, and to deduct the amount of copper precipitated from the total quantity apparently reduced by the sugar. From 23 analyses the author finds that 100 parts of maltose correspond to 114.2 of copper. (3) The reaction between maltose and Fehling's solution is complete in 2 minutes; in the case of impure specimens of maltose, therefore, this time should not be exceeded. (4) In order to destroy certain organic substances mixed with the cuprous oxide, the tube should be heated before it is reduced in the stream of hydrogen.

F. H. L.

Urine reducing Fehling Solution. Crolas. (*Répertoire de Pharm.*, Aug., 1896, p. 367; through *Ann. de Chimie Analyt.*, i. [19], 370.)—The author finds that the urine of persons who have been eating asparagus reduces Fehling's solution, though it contains no glucose.

C. S.

INORGANIC ANALYSIS.

Estimation of Zinc in Organic Salts. G. v. Ritter. (*Zeit. anal. Chem.*, 1896, xxxv., 311-314.)—The estimation of zinc in organic compounds by precipitation as sulphide or carbonate is, in the author's opinion, too troublesome and requires too much attention to detail. This is obviated in his method, which consists in treating the zinc salt with concentrated nitric acid, evaporating the acid and igniting the residue, when the zinc is left as oxide. The tabulated results of the analyses of a number of organic zinc salts closely correspond with those required by theory. A porcelain crucible should be used for the ignition, since platinum is rapidly attacked. Experiments in which sulphuric acid was substituted for nitric acid gave results several per cent. too high.

C. A. M.

Sources of Error in Volhard's and Similar Methods of Determining Manganese in Steel. G. Auchy. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 498-511.)—The sources of error in Volhard's process as indicated by the author's experiments are: (1) Incomplete neutralization with zinc oxide, usually giving high results. (2) Too sudden addition of the necessary excess of zinc oxide frequently giving low results. (3) The titration in nitric acid solution giving results 0.01 or 0.02 per cent, too high. (4) Neutralization by zinc oxide in hot solution giving high results.

The author prefers Stone's modification as being easier and quicker, the evaporation with sulphuric acid being dispensed with, and the precipitated ferric oxide rapidly subsiding in the nitric acid solution. In the following outline of his process the necessary precautions are printed in italics. Three and three-tenths grammes of drillings are dissolved in 50 c.c. of nitric acid (specific gravity 1.20) and washed into a 500 c.c. measuring flask. Two-thirds of the amount of sodium carbonate solution necessary for complete neutralization are added, and the *liquid cooled*. Zinc oxide emulsion is then added until the solution stiffens, *an excess being avoided*. After dilution to about three-fourths the capacity of the flask the whole is allowed to stand until the ferric oxide begins to settle, and a *considerable excess of zinc oxide emulsion then added* to the colourless solution. After being made up to the mark and well shaken the precipitate is allowed to settle, and 250 c.c. of the clear solution heated in a flask to boiling and titrated with permanganate of strength 0.0056. After making the necessary deductions for impurities in the sodium carbonate and zinc oxide, the number of c.c. of permanganate taken is divided by 10, and 0.02 *per cent. deducted* from the result.

C. A. M.

Note on the Solubility of Bismuth Sulphide in Sodium Sulphide. T. B. Stillman. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 683, 684.)—The author proves that in the ordinary method of separating lead, copper, and bismuth from arsenic, antimony, and tin, by dissolving the sulphides of the latter metals in sodium sulphide, a considerable proportion of bismuth sulphide also passes into solution. In an experiment with 0.128 gramme of pure bismuth nitrate, the amount of sulphide dissolving in sodium sulphide was 0.031 gramme; and with an alloy containing lead, copper, antimony, tin, and a very small amount of bismuth, the whole of the bismuth was dissolved. Therefore, if one gramme of an alloy were taken for the analysis, and sodium sulphide used as the separating agent, as much as 3 per cent. of bismuth might be present, and none of it be left as an insoluble sulphide with the lead sulphide.

C. A. M.

Estimation of Zinc in Iron Ores. Kinder. (*Stahl u. Eisen*, 1896, xvi., 675; through *Chem. Zeit. Rep.*, 1896, 242.)—Five grammes of the zinciferous ore are dissolved in hydrochloric acid, and 20 or 25 c.c. of 1:2 sulphuric acid added to precipitate the lead. The excess of acid is evaporated off, the mass taken up in water, filtered, and the liquid saturated with sulphuretted hydrogen. Any copper sulphide is removed, 25 c.c. of a solution of ammonium formate and 15 c.c. of formic acid introduced, and the zinc sulphide collected on a filter. If the latter is dark in colour, it is dissolved in hydrochloric acid, neutralized with ammonia, more formic

acid added, and reprecipitated with sulphuretted hydrogen. The sulphide may either be weighed as such, or dissolved in acid, thrown down with sodium carbonate, and converted into oxide.

F. H. L.

The Delicacy of certain Tests for Metals. B. Neumann. (*Chem. Zeit.*, 1896, xx., 768.)—The author has collected the statements made by different investigators respecting the delicacy of most of the usual reactions employed in testing for metals, which, with the results of a number of experiments on this subject carried out by himself, he has arranged in tabular form.

F. H. L.

Critical Examination of the Volumetric Methods of Estimating Nickel. Goutal. (*Ann. de Chimie Analyt.*, i., [16], 305.)—The author draws the following conclusions from his investigations into the various methods proposed for estimating nickel volumetrically: (1.) The volumetric method, indicated in Mohr's work and recommended by A. Carnot, is based on the employment of bromine and potassium cyanide. It is well adapted for the determination of nickel in ores and metallurgical products when there is a sensible quantity of nickel present, even in association with cobalt. It offers considerable advantages on account of its simplicity and accuracy.

(2.) Th. Moore's method of direct titration with potassium cyanide is useful, especially in the study of metallurgical products of low percentage and containing merely traces of cobalt. Good results are obtained, provided the amount of silver nitrate in the cyanide solution be varied in inverse proportion to the quantity of nickel in the substance. Cobalt should be removed before the titration is performed, and manganese, copper, zinc and iron should also be separated, or the results may be prejudiced. The liquid should be only slightly alkaline, and not contain too large a quantity of ammonium salts, and the temperature must not exceed 20° C.

(3.) The phosphate, ammonium molybdate or nitroso-naphthol methods of estimation, which are generally performed after the separation of cobalt, are more difficult, and slower, but are very reliable in experienced hands.

(4.) The other volumetric methods proposed appear to be less accurate, and do not offer any apparent advantages over the gravimetric processes.

C. S.

Normal Sodium Oxalate in Volumetric Analysis. S. P. L. Sørensen. (*Tidsskrift Physik og Cheme.*, 1896, i., 172; through *Chem. Zeit. Rep.*, 1896, 235.)—To prepare pure normal sodium oxalate, the commercial product should be dissolved in water, caustic soda added to faint alkalinity, the liquid evaporated to one-tenth its bulk, the salt removed, powdered, washed, and re-crystallized. It is an anhydrous body, can be dried at 125° to 160° C., and it is not hygroscopic. In order to standardize volumetric acids, a portion of the oxalate may be weighed off, converted into carbonate by gentle ignition, and a titration carried out as usual. It is also better adapted for checking the strength of permanganate than oxalic acid itself.

F. H. L.

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